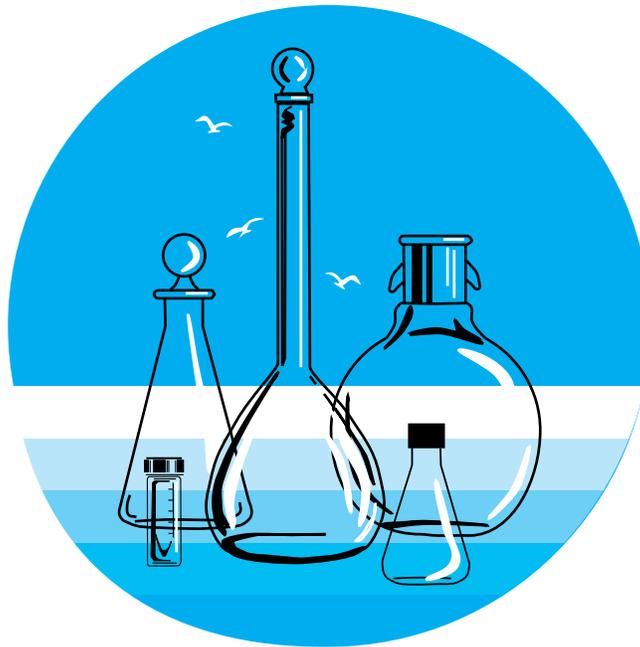


**National Status and Trends Program**  
for Marine Environmental Quality

**Sampling and Analytical Methods of the  
National Status and Trends Program  
National Benthic Surveillance and Mussel Watch Projects  
1984-1992**

**Volume I**

**Overview and Summary of Methods**



Silver Spring, Maryland  
July, 1993

**noaa** NATIONAL OCEANIC AND ATMOSPHERIC ADMINISTRATION

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Coastal Monitoring and Bioeffects Assessment Division  
Office of Ocean Resources Conservation and Assessment  
National Ocean Service



NOAA Technical Memorandum NOS ORCA 71

Sampling and Analytical Methods of the  
National Status and Trends Program  
National Benthic Surveillance and Mussel Watch Projects  
1984-1992

Volume I

Overview and Summary of Methods

G. G. Lauenstein and A. Y. Cantillo  
(Editors)

Silver Spring, Maryland  
July, 1993

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Ronald H. Brown  
Secretary

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D. James Baker  
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**Analysis of Marine Sediment and Bivalve Tissue by X-Ray Fluorescence, Atomic Absorption and Inductively Coupled Plasma Mass Spectrometry**

E. Crecelius, C. Apts, L. Bingler, O. Cotter, S. Kiesser and R. Sanders

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C. Peven, A. Uhler and D. West

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**Standard Analytical Procedures of the NOAA National Analytical Facility,  
1985-1986 (Revised). Extractable Toxic Organic Compounds**

W. D. MacLeod Jr., D. W. Brown, A. J. Friedman, D. G. Burrows, O. Maynes, R. W. Pearce, C. A. Wigren and R. G. Bogar

Revised/Edited by A. Cantillo, C. Sloan, and G. Lauenstein

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C. A. Sloan, N. G. Adams, R. W. Pearce, D. W. Brown, and S. - L. Chan

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C. S. Peven and A. D. Uhler

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**NIST Methods for Certification of SRM 1941 and SRM 1974**

M. M. Schantz, B. A. Benner, Jr., S. N. Chesler, R. G. Christensen, B. J. Koster, J. Kurz,  
R. M. Parris, and S. A. Wise

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## FOREWORD

Management decisions on the use of the resources of the nation's coastal areas require reliable, high quality information on the status and trends of environmental quality conditions in those areas. Since 1984, the National Oceanic and Atmospheric Administration (NOAA) has provided this information through its National Status and Trends (NS&T) Program. The program's objectives include defining the geographic distribution of contaminant concentrations in tissues of marine organisms and in sediments, and documenting biological responses to contamination. Samples have been collected since 1984 by the NS&T Program's National Benthic Surveillance Project, and since 1986 by the Mussel Watch Project. The National Benthic Surveillance Project has been a cooperative effort between two NOAA organizations, the National Ocean Service and the National Marine Fisheries Service. Mussel Watch Project sample collection and analyses have been performed by contract laboratories and the National Ocean Service.

Contaminant measurements made by the NS&T Program must be of the highest quality for NOAA to meet its statutory and scientific responsibilities that require data for modeling, assessment, prediction, and management. To meet these data requirements, quality assurance protocols have been an integral part of the NS&T Program since its inception. Documentation of sampling and analytical methods are an essential part of quality assurance practices. This document provides NS&T data users the information necessary to determine the quality of the data generated by the program and its potential comparability to other data sets.

Charles N. Ehler  
Director  
Office of Ocean Resources Conservation and Assessment

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## ACRONYMS AND DEFINITIONS

AHH	Aryl hydrocarbon hydroxylase
AH	Aromatic hydrocarbons
Alumina	Used as an adsorbent in liquid-solid chromatography made by dehydrating alumina trihydrate
Aqua regia	A mixture of concentrated HNO <sub>3</sub> and HCl
BCR	Community Bureau of Reference
BHC	1,2,3,4,5,6-Hexachlorocyclohexane. Lindane is gamma-BHC or -HCH
CAS	Chemical Abstract Service
CH <sub>2</sub> Cl <sub>2</sub>	Methylene chloride or dichloromethane
CHN analyzer	Carbon-hydrogen-nitrogen analyzer
Clean room	A controlled laboratory environment which minimizes sample contamination
COLOR	Colorimetry
CRM	Certified Reference Material produced by the National Research Council of Canada, BCR or other organization
CVAA	Cold vapor atomic absorption spectrometry
DB5	Capillary column, internal coating 5% phenyl silicone and 95% methyl silicone
DDT	1,1,1-Trichloro-2,2-bis[ <i>p</i> -chlorophenyl]ethane
ECD	Electron capture detector
EPA	U.S. Environmental Protection Agency
FAA	Flame atomic absorption spectrometry
FID	Flame ionization detector
FD	Fluorescence detector
FTM	Fluid thioglycollate medium
GC	Gas chromatography
GERG	Geochemical and Environmental Research Group, Texas A&M University
GFAA	Graphite furnace atomic absorption spectrometry
HCH	1,2,3,4,5,6-Hexachlorocyclohexane. Lindane is gamma-HCH
HEPA	High efficiency particulate attenuator
HAA	Hydride generation atomic absorption spectrometry
H <sub>3</sub> BO <sub>3</sub>	Boric acid
HCB	Hexachlorobenzene
HCl	Hydrochloric acid
HF	Hydrofluoric acid
HNO <sub>3</sub>	Nitric acid
HP	High purity
HPLC	High performance liquid chromatography
ICES	Intergovernmental Commission for the Exploration of the Seas
ICP-MS	Inductively coupled plasma - mass spectrometry
IUPAC	International Union of Pure and Applied Chemistry
Kynar	Poly(vinylidene fluoride), a form of Teflon®
LOC	Level of chlorination
LOD	Limit of detection
LOQ	Limit of quantitation
MDL	Method detection limit
MFO	Mixed function oxidase
MS	Mass spectrometry
MWP	Mussel Watch Project, National Status and Trends Program
NAA	Neutron activation analysis

NAF	National Analytical Facility of NOAA's National Marine Fisheries Service, Seattle, Washington; now incorporated into NWFSC
Na <sub>2</sub> SO <sub>4</sub>	Sodium sulfate
NBSP	National Benthic Surveillance Project, National Status and Trends Program
NEFSC	NOAA/NMFS/Northeast Fisheries Science Center
NIST	National Institute of Standards and Technology (formerly National Bureau of Standards)
NMFS	National Marine Fisheries Service, NOAA
NOAA	National Oceanic and Atmospheric Administration, Department of Commerce
NOS	National Ocean Service, NOAA
NS&T	National Status and Trends Program
NWFSC	NOAA/NMFS/Northwest Fisheries Science Center
PAH	Polycyclic aromatic hydrocarbons
PCB	Polychlorinated biphenyls
RM	Reference or Research Material
RPM	Revolutions per minute
SAIC	Science Applications International Corporation, Inc.
SEFSC	NOAA/NMFS/Southeast Fisheries Science Center
Sephadex LH-20	A controlled porosity gel used in liquid-solid chromatography
Silica gel	Silicon dioxide beads used in column chromatography to separate organic compounds
SIM	Selected ion monitoring
SOP	Standard Operating Procedure
SRM	Standard Reference Material, produced by the National Institute of Standards and Technology
TAMU	Texas A&M University
TBT	Tributyltin
XRF	X-ray fluorescence spectrometry

# NOAA National Status and Trends Program Development and Methods

G. G. Lauenstein, A. Y. Cantillo and S. S. Dolvin  
Coastal Monitoring and Bioeffects Assessment Division  
Office of Ocean Resources Conservation and Assessment  
National Ocean Service

## ABSTRACT

The quantification of environmental contaminants by the National Oceanic and Atmospheric Administration's National Status and Trends Program began in 1984. Polycyclic aromatic hydrocarbons, butyltins, polychlorinated biphenyls, DDT (its metabolites and other chlorinated pesticides), and trace and major elements were quantified in estuarine and coastal sediment and tissue samples. This NS&T Program has two major monitoring components. The National Benthic Surveillance Project is responsible for quantification of contamination in fish tissue and sediments, and for developing and implementing new methods to define the biological significance of environmental contamination. The Mussel Watch Project monitors contaminant concentrations by quantifying chemicals in bivalve mollusks and sediments. Field sampling procedures and analytical quantification methods of the National Benthic Surveillance and Mussel Watch Projects are described in this document. The evolution of analytical methods, and the quality assurance/control protocols are also discussed. Program development, quality assurance, and summaries of field and analytical methods are found in Volume I. Volumes II through IV provide detailed information, on a laboratory by laboratory basis, about complementary methods, elemental analyses, and organic analyses, respectively.

## 1. INTRODUCTION

In response to the need for information on effects of human activities on environmental quality in coastal and estuarine areas, and the need to develop management strategies to deal with these conditions, the National Oceanic and Atmospheric Administration (NOAA) initiated in 1984, the National Status and Trends (NS&T) Program for Marine Environmental Quality. The purpose of this program is to determine the current status and detect changes in the environmental quality of our Nation's estuarine and coastal waters. Field sampling procedures, analytical chemistry methods, histopathology and complementary measurements of the NS&T Program's two monitoring projects, the National Benthic Surveillance Project (NBSP) and the Mussel Watch Project (MWP), are discussed in this document.

The NBSP collects and analyzes benthic fish and sediments from sites around the coastal and estuarine United States, including Alaska. This effort is primarily performed by NOAA's National Marine Fisheries Service (NMFS). The MWP collects and analyzes bivalve mollusks and associated sediments from around the United States, including the Great Lakes, Alaska, Hawaii, and Puerto Rico. This effort is administered by NOAA, with collection and analyses being performed under contract. From 1984 through 1994, the Geochemical and Environmental Research Group, Texas A&M University (TAMU), College Station, TX has collected and analyzed samples from the Gulf Coast. During this time Battelle Memorial Institute, Duxbury, MA, and Sequim, WA has collected and analyzed samples from the U.S. East and West Coasts, including sites in the Hawaiian Islands and Alaska. During 1986-1989 samples from along the California and Hawaiian coasts were collected and analyzed by Science Applications International Corporation, Inc. (SAIC).

The Quality Assurance (QA) Project of the NS&T Program assures that despite variations in the analytical methodologies used in the monitoring projects, data are comparable between all laboratories. The QA Project has not been limited to NS&T Program laboratories, but has been made available to other laboratories quantifying estuarine and coastal contamination. Since 1990, the Environmental Protection Agency's (EPA's) Environmental Monitoring and Assessment Program-Estuarines (EMAP-E) has been a participant and contributor to the QA Project.

The NS&T Program does not prescribe specific analytical methods but encourages the use of state-of-the-art procedures. This technical memorandum documenting the evolution of sampling and analytical procedures was prepared from existing and new reports from the NOAA/NMFS, responsible for the NBSP; and NOAA's MWP contractors: Battelle, TAMU, and SAIC. Because readers may need different levels of specificity, the main body of the document summarizes the methods used by all NS&T Program laboratories while Volume II Complementary Measurements, Volume III Elemental Analytical Methods, and Volume IV Trace Organic Analytical Methods describe in detail the analytical methodologies used by each NS&T laboratory.

## 2. DEVELOPMENT OF THE NS&T PROGRAM

In October 1983, the NS&T Program organized a meeting of marine scientists from government, academia, and the private sector to discuss the feasibility of performing estuarine and coastal environmental monitoring on a nationwide basis. Contaminants and sample matrices selected for quantification by the NS&T Program were defined at this workshop (Boehm, 1983). The general characteristics of contaminants to be quantified were that they: should have a demonstrated health risk; should have been released into the environment in significant quantities so they are measurable; should have long half-lives once released; and should have a high potential for bioaccumulation. The consensus was that chemicals meeting those criteria were: polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), chlorinated pesticides, and trace elements. In addition, total organic carbon, coprostanol, iron, and aluminum for sediment; and total lipid content for animal tissues; were suggested for normalizing the data and were incorporated into the NS&T Program.

### 2.1. Selection of elements and compounds

Within these guidelines, chemicals to be quantified were selected in part from EPA's Priority Pollutant List (Keith and Telliard, 1979). Tables I.1 - I.5 list the analytes and analyte groups quantified by the NS&T Program, and whether these are on the EPA list. Twelve of the 13 trace elements defined in the 1983 EPA list were incorporated into the NS&T Program with the exception of beryllium. Of the 24PAHs quantified by the NS&T Program, 16 are on the EPA list. The chlorinated pesticides were primarily selected from the EPA list or are closely related contaminants. Although the EPA suggested quantifying PCBs by comparison to Aroclor standards, the NS&T Program began quantifying PCBs by congener and extrapolating to chlorination levels.

### 2.2. Recommended detection limits

The 1983 workshop committee recommended detection limits for the quantification of trace elements and organics (Table A.1, Appendix A). In 1989, when NOAA published a request for proposals for the 1990-1994 portion of the MWP, the suggested detection limits were the minimum level of technical expertise required of analytical chemists (Tables A.2 through A.4, Appendix A).

Table I.1. Major and trace elements determined in the NS&T Program since 1984.

Symbol	Element	Symbol	Element	Symbol	Element
Al	Aluminum	Cu	Copper $\Delta\Diamond$	Ag	Silver $\Delta\Diamond$
Si	Silicon	Zn	Zinc $\Delta\Diamond$	Cd	Cadmium $\Delta\Diamond$
Cr	Chromium $\Delta$	As	Arsenic $\Delta$	Hg	Mercury $\Delta$
Mn	Manganese	Se	Selenium $\Delta$	Tl	Thallium $\Delta$
Fe	Iron	Sn	Tin	Pb	Lead $\Delta\Diamond$
Ni	Nickel $\Delta\Diamond$	Sb	Antimony $\Delta$		

$\Delta$  Trace elements contained in the EPA Priority Pollutants List (Keith and Telliard, 1979).

$\Diamond$  Trace elements quantified by both the NS&T Program and the earlier EPA Mussel Watch Program (1976-1978).

Table I.2. Polycyclic aromatic hydrocarbons determined in the NS&T Program since 1984.

Analytes	CAS Numbers $\Diamond$	Analytes	CAS Numbers $\Diamond$
Acenaphthene $\Delta$	83-32-9	2,6-Dimethylnaphthalene	581-42-0
Acenaphthylene $\Delta\uparrow$	208-96-8	Fluoranthene $\Delta$	206-44-0
Anthracene $\Delta$	120-12-7	Fluorene $\Delta$	86-73-7
Benz[ <i>a</i> ]anthracene $\Delta$	56-55-3	Indeno[1,2,3- <i>cd</i> ]pyrene $\Delta\uparrow$	193-39-5
Benzo[ <i>a</i> ]pyrene $\Delta$	50-32-8	1-Methylnaphthalene	90-12-0
Benzo[ <i>e</i> ]pyrene	192-97-2	2-Methylnaphthalene	91-57-6
Benzo[ <i>b</i> ]fluoranthene $\Delta\uparrow$	205-99-2	1-Methylphenanthrene	832-69-9
Benzo[ <i>k</i> ]fluoranthene $\Delta\uparrow$	207-08-9	Naphthalene $\Delta$	91-20-3
Benzo[ <i>ghi</i> ]perylene $\Delta\uparrow$	191-24-2	Perylene	198-55-0
Biphenyl	92-52-4	Phenanthrene $\Delta$	85-01-8
Chrysene $\Delta$	218-01-9	Pyrene $\Delta$	129-00-0
Dibenz[ <i>a,h</i> ]anthracene $\Delta$	53-70-3	1,6,7-Trimethylnaphthalene $\uparrow$	2245-38-7

$\Delta$  Organic contaminants contained in the EPA Priority Pollutants List (Keith and Telliard, 1979).

$\Diamond$  Chemical Abstracts Service Registry Numbers.

$\uparrow$  Compounds added since 1988.

Table I.3. Chlorinated pesticides determined in the NS&T Program since 1984.

Analytes	CAS Numbers <sup>◇</sup>	Analytes	CAS Numbers <sup>◇</sup>
Aldrin <sup>Δ</sup>	309-00-2	Dieldrin <sup>Δ</sup>	60-57-1
<i>cis</i> -Chlordane <sup>Δ</sup>	5103-71-9	Endrin <sup>Δ†</sup>	72-20-8
2,4'-DDD	53-19-0	Heptachlor <sup>Δ</sup>	76-44-8
4,4'-DDD <sup>Δ</sup>	72-54-8	Heptachlor epoxide <sup>Δ</sup>	1024-57-4
2,4'-DDE	3424-82-6	Hexachlorobenzene	118-74-1
4,4'-DDE <sup>Δ</sup>	72-55-9	gamma-HCH <sup>Δ</sup>	58-89-9
2,4'- DDT	58633-27-5	Mirex	2385-85-5
4,4'-DDT <sup>Δ</sup>	50-29-3	<i>trans</i> -Nonachlor	39765-80-5

<sup>Δ</sup> Organic contaminants contained in the EPA Priority Pollutants List (Keith and Telliard, 1979).

<sup>†</sup> Was quantified in the program through 1986.

<sup>◇</sup> Chemical Abstracts Service registry numbers.

### 2.3. Analytical definition of detection limits

When the program began, data at or near the detection limit were to be reported following procedures defined by Keith *et al.* (1983) who defined the limit of detection (LOD) as the lowest concentration level that can be determined to be statistically different from a blank. The standard deviation (used to determine the LOD) was defined by replicate measures of the difference between the lowest concentration of analyte instrumentally detectable and a blank value (Freitas *et al.*, 1989). Any measured value below the LOD were considered to be not detected (Table I.6). The limit of quantitation (LOQ) was defined as 10x the standard deviation of the blank (Table I.6). Data reported between the LOD and LOQ were given a footnote in the NS&T database.

In 1990, the NS&T Program replaced LODs and LOQs with Method Detection Limits (MDLs). These values are not based on the variability of blanks but rather on the standard deviation of the signal from replicate analysis of real matrix samples containing, in principle, low levels of the analyte (CFR, 1990). The MDL is "x" times the standard deviation, where "x" is defined by the Student's t-distribution to cover 99% of the distribution of possible values (for 7 analyses,  $x = 3.5$ ). MDLs were developed from a minimum of 7 replicate analyses. Tables A.5 through A.46 (Appendix A) present the "detection limits" for the NBSP and MWP laboratories for all analytes quantified by the NS&T Program from 1984 through 1991. Detection limits, until 1990, for the NBSP<sup>□</sup> laboratories were the lowest reported values from a given year's data unless otherwise noted. The detection limits for the East and West Coast MWP laboratories, until 1990, were instrumental detection limits based on the "noise" in analysis of spiked blanks, while the Gulf Coast detection limits starting in 1989, are MDLs as defined above.

<sup>□</sup> The actual detection limit for an individual analyte in a sample depends on factors such as the procedure used to analyze the sample, the sample weight, the percent dry weight, the smallest GC peak area of any detected analyte in the appropriate GC calibration solution with the lowest concentration analyzed with the sample, and the GC detector response to the individual analyte relative to the GC internal standard. Approximate 1993 detection limits for NBSP sediments based on a 10 g sample size and a 60% dry weight are 0.2 to <2 ng/g for chlorinated hydrocarbons and 2 to <8 ng/g for PAHs. The approximate 1993 detection limits for livers based on a 3 g sample size and a 30% dry weight are 0.5 to <5 ng/g for chlorinated hydrocarbons. Stomach contents detection limits for a sample of 3 g and 20% dry weight were 0.5 to <5 ng/g for chlorinated hydrocarbons and 0.3 to <2 ng/g for aromatic hydrocarbons; C. Sloan, NOAA/NMFS/NWFSC, Seattle, WA, personal communication, 1993.

Table I.4. Polychlorinated biphenyls determined in the NS&T Program since 1984.

Chlorination Groups <sup>Δ</sup>		
Dichlorobiphenyls		Hexachlorobiphenyls
Trichlorobiphenyls		Heptachlorobiphenyls
Tetrachlorobiphenyls		Octachlorobiphenyls
Pentachlorobiphenyls		Nonachlorobiphenyls
Individual congeners	IUPAC Numbers	CAS registry numbers <sup>◇</sup>
2,4'-Dichlorobiphenyl	8	34883-43-7
2,2',5'-Trichlorobiphenyl	18	37680-65-2
2,4,4'-Trichlorobiphenyl	28	7012-37-5
2,2',3,5'-Tetrachlorobiphenyl	44	41464-39-5
2,2',5,5'-Tetrachlorobiphenyl	52	35693-99-3
2,3',4,4'-Tetrachlorobiphenyl	66	32598-10-0
3,3',4,4'-Tetrachlorobiphenyl	77(110*)	32598-13-3 (38380-03-9)
2,2',4,5,5'-Pentachlorobiphenyl	101	37680-73-2
2,3,3',4,4'-Pentachlorobiphenyl	105	32598-14-4
2,3',4,4',5-Pentachlorobiphenyl	118	31508-00-6
3,3',4,4',5-Pentachlorobiphenyl	126	57465-28-8
2,2',3,3',4,4'-Hexachlorobiphenyl	128	38380-07-3
2,2',3,4,4',5'-Hexachlorobiphenyl	138	35065-28-2
2,2',4,4',5,5'-Hexachlorobiphenyl	153	35065-27-1
2,2',3,3',4,4',5-Heptachlorobiphenyl	170	35065-30-6
2,2',3,4,4',5,5'-Heptachlorobiphenyl	180	36065-29-3
2,2',3,4',5,5',6-Heptachlorobiphenyl	187	52663-68-0
2,2',3,3',4,4',5,6-Octachlorobiphenyl	195	52663-78-2
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	206	40186-72-9
2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl	209	2051-24-3

<sup>Δ</sup> Organic contaminants contained in the EPA Priority Pollutants list (Keith and Telliard, 1979). Beginning in 1988, individual congener concentrations rather than chlorination groups began being quantified.

<sup>◇</sup> Chemical Abstracts Service registry numbers.

\* These coelute on the GC column type used for these analyses and are discussed in detail in section 5.2.1.1.

Table I.5. Organometallic compounds determined in the NS&T Mussel Watch Project. <sup>Δ</sup>

Organotins <sup>Δ◇</sup>	CAS Numbers*
Monobutyltin trichloride	1118-46-3
Dibutyltin dichloride	683-18-1
Tributyltin chloride	1461-22-9
Tetrabutyltin	1461-25-2

<sup>Δ</sup> Measurement of organotins on a national basis began in 1989.

<sup>◇</sup> Only the cation portion of the molecule is quantified because many anions can combine with the tin-containing cation. Tributyltin is the primary biocide; dibutyltin and monobutyltin are metabolites of tributyltin; and tetrabutyltin is an unintended manufacturing by-product.

\* Chemical Abstracts Service registry numbers.

Table I.6. Analytical definition of detection limits (Keith *et al.*, 1983).

Analyte concentration in units of SD	Degree of reliability	Reported As
< 3	Region of questionable detection	ND
3	Limit of Detection (LOD)	LOD
3 to 10	Region of less-certain quantitation	<LOQ
10	Limit of quantitation	LOQ
> 10	Region of quantitation	Value

SD - Standard deviation in measurement at instrumental detection limit.

Detection limits (LODs) based on spiked blanks are not the same as MDLs based on actual samples such as fish, mollusk tissues, or sediments. It is because the NS&T Program changed to defining detection limits as the MDL that a number of the detection limits appear to be higher for 1990 and afterward. At very low contaminant concentrations, the relative variance of an analysis increases as the actual concentration decreases. If the matrix being used to define the detection limit has any contaminants that are somewhat elevated with respect to background the resulting detection limit, based on the absolute variance, can be higher than concentrations at which contaminants can actually be quantified. It is difficult, however, to find a reference material that is low in all the contaminants quantified by the NS&T Program. In these situations, detection limits calculated using MDLs result in higher detection limits than those limits derived from spiked blanks.

#### 2.4. Recommended number of replicates

The 1983 workshop recommended compositing of samples and replication of stations at each site. The ability to detect differences is limited by environmental and analytical variability, and one way to minimize sample variability is to composite samples at stations. Compositing also provides added material for the chemist. Major and trace element variability can be accounted for by using a single composite sample of 5-10 organisms (Lobel *et al.*, 1991a). It is for this reason that no fewer than 20 mollusks are collected at any MWP station. In the NS&T Program, variability of concentrations measured at a single site in a given year was based on the analysis of 3 separate composite samples (stations).

#### 2.5. Recommended sample matrices

##### 2.5.1. Sediments

All of the elements and organic contaminants measured by the NS&T Program readily adsorb on to particle surfaces and are generally concentrated in fine grained sediments. Thus, these chemicals tend to accumulate in areas of fine sediment deposition within estuaries and on adjacent continental shelves. Analysis of sediments is used to compare levels of contamination among sites. Also, since large amounts of sediment contaminant data exist from other monitoring programs, the NS&T data could be put into a broader context to further quantify

national sediment contamination. The disadvantage of sediment analysis is that it does not provide a reliable basis for identifying temporal trends in levels of contamination. Data on rates of sediment deposition and mixing are almost never available but are required to know the time range represented by the surface sample. The advantage of characterizing sediments is that unlike organisms, species intercomparability problems do not exist. Even the most cosmopolitan of sentinel species are not found in all coastal and estuarine areas quantified by the NS&T Program.

#### 2.5.2. Bivalves

Prior to the initiation of the NS&T Mussel Watch Project, mollusks had already been used by the U.S. EPA, California Mussel Watch, and the international scientific community. Bivalve mollusks were selected as a primary sample matrix because there was evidence that their tissues can change in response to the environment in a matter of months (Roesijadi *et al.*, 1984; Sericano, 1993). The 1983 workshop participants and others defined some of the advantages of using mollusk bivalves for environmental monitoring (Berner *et al.*, 1976; Farrington *et al.*, 1980; Farrington, 1983; and Tripp and Farrington, 1984):

- Bivalves are cosmopolitan, thus minimizing the problems inherent in comparing data from markedly different species;
- Bivalves are sessile and thus better than mobile species as integrators of contaminants in a given area;
- A correlation is expected between the contaminant content of the organisms and the average contaminant concentration in the surrounding water;
- Bivalves have high tolerances to many contaminants in comparison to fish and crustaceans and so survive in adverse environmental conditions;
- Bivalves are able to bioconcentrate chemicals by factors of  $10^2$  to  $10^5$  from seawater making analytical detection easier;
- Bivalves appear to have minimal ability to metabolize petroleum hydrocarbons and chlorinated hydrocarbons, including PCBs and DDTs;
- Bivalves have relatively stable, local populations that are extensive enough to be sampled repeatedly, providing data on short and long-term temporal changes in the concentrations of contaminants; and
- Bivalves are commercially valuable seafood species on a world-wide basis, therefore, measurement of chemical contamination is of interest for public health considerations.

Because of these advantages, the NS&T Program included the "mussel watch" concept as a major program element.

#### 2.5.3. Fish

Fish samples were also incorporated in the NS&T Program primarily because of their ability to bioaccumulate contaminants (Boehm, 1983). A disadvantage of using fish for monitoring purposes is that fish are not sessile and some species collected by the NS&T Program are not resident, on a year round basis, in the body of water being characterized. Contaminants found in their tissues may have been accumulated in another body of water.

#### 2.5.4. Other species

At the 1983 workshop, use of birds and mammals in the NS&T Program was also discussed but their use was not adopted because of their ability to metabolize organic contaminants, their wide range of movement, and in the case of birds, difficulty in ascribing chemical levels to marine sources alone (Boehm, 1983).

#### 2.5.5. Seawater

Seawater chemical analyses were not included in the monitoring effort because:

"while seawater represents the primary medium for transport of pollutants and transfer of pollutants to the biota, and while most of the existing marine toxicological data relates to levels of aqueous phase pollutants (i.e. water quality criteria), actual pollutant levels in seawater are highly variable due to the sporadic effects of runoff from storms, dumping events, etc. Most water column pollutant measurements in estuaries or coastal waters represent snapshots of data. Therefore, this variability, coupled to the fact that pollutant levels may be quite low in the samples, leads to the recommendation that direct analysis of seawater for pollutant levels not be included in the Status and Trends Program." (Boehm, 1983)

### 3. SAMPLING PROCEDURES

Site designation and sample collection differ to some extent between the NBSP (Section 3.1) and the MWP (Section 3.2). Sampling for the NBSP is almost exclusively conducted from ships and boats because fish are primarily collected by trawling. Mussel Watch Project mollusks are collected from shore at intertidal sites and subtidal sites were primarily collected from boats.

#### 3.1. National Benthic Surveillance Project

The NS&T NBSP began in 1984 and has collected fish and associated sediment samples on an annual basis since that time. The NBSP is a cooperative effort between three NOAA elements: the National Ocean Service's Office of Ocean Resources Conservation and Assessment, the NOAA Corps, and NMFS. In 1984, samples were collected and analyzed from 50 sites around the United States, including Alaska, and by 1990, 149 sites had been sampled (Lauenstein *et al.*, 1993). The actual field collections and laboratory analysis are performed by NMFS. For the years 1984-1986, three laboratories were responsible for NBSP activities. The Northeast Fisheries Science Center (NEFSC) collected samples from the Chesapeake Bay northward to Maine. The Southeast Fisheries Science Center (SEFSC) collected samples from Pamlico Sound southward and along the coast of the Gulf of Mexico. The Northwest Fisheries Science Center (NWFSC) was responsible for sample collection from the West Coast states (California, Oregon, Washington, and Alaska). In 1987, the NWFSC assumed responsibility for the collection of samples from the northeast Atlantic Coast. Individual laboratory areas of responsibility are shown in Table I.7.

Once sampling in a certain geographic area is initiated, repeat sampling occurs during the same time frame. Northeast samples (Chesapeake Bay through Maine) have been collected during March and April. Southeast samples have been collected from August to October. Gulf Coast samples have been collected from August to October. West Coast samples have been taken from

Table I.7. Laboratories analyzing National Benthic Surveillance Project samples.

Trace Elements	1984-1986	1987	1988-1993
Northeast Coast	NEFSC	NWFSC	SEFSC
Southeast and Gulf Coasts	SEFSC	SEFSC	SEFSC
West Coast	NWFSC	NWFSC	NWFSC
Organic Contaminants	1984-1986	1987	1988-1993
Northeast Coast	NEFSC	NWFSC	NWFSC
Southeast and Gulf Coasts	SEFSC	SEFSC	NWFSC
West Coast	NWFSC	NWFSC	NWFSC

NEFSC - NOAA/NMFS/Northeast Fisheries Science Center, Sandy Hook, NJ, for trace elements, and Gloucester, MA, for organics.

SEFSC - NOAA/NMFS/Southeast Fisheries Science Center, Beaufort, NC, for trace elements, and Charleston, SC, for organics.

NWFSC - NOAA/NMFS/Northwest Fisheries Science Center, Seattle, WA, for organics and trace elements.

May through July. Alaska samples have been taken from May to August. The collection of fish is not directly tied to their spawning cycle, though different age classes may be found in certain estuaries during different times of the year.

Samples are collected directly from NOAA ships or from small boats launched from NOAA ships, or on occasion from chartered vessels or ships-of-opportunity. The vessel primarily used on the East and Gulf Coasts is the NOAA Ship FERREL. West Coast and Alaska work is performed aboard the NOAA Ship McARTHUR. NOAA research vessels are under the direction of the NOAA Corps.

### 3.1.1. Site designation

As discussed in Section 2.4, three stations comprise an NS&T Program site. Specific site information for where fish and sediments were collected for the NBSP are listed in Table A.47 (Appendix A). Fish are not sessile and so, over the years, fish trawls have been made along different tracks in the water body of interest. More than one trawl may be required to capture enough fish of the right size range for monitoring purposes. Therefore, a nominal site center has been defined for all NBSP sites as an area 2 km in diameter and is revisited for sample collection once it has been defined. Each NBSP site bears both location and site names. The location name refers to a general location (e.g., Boston Harbor). The site name defines a more specific location within the designated geographic area (e.g., Boston Harbor, Deer Island). The site location and specific site name are then used to develop a unique site acronym (e.g., BOSDI for the Deer Island site in Boston Harbor). More detailed site information can be found in Lauenstein *et al.*, 1993.

### 3.1.2. Sediments

Sediments were collected concurrently with fish specimens at each NBSP site. Sediment samples were analyzed for selected organic compounds, trace elements, total organic carbon, moisture content, and particle size distribution.

#### 3.1.2.1. Sediment collection

Sediment samples were obtained using a specially constructed box corer or a standard Smith-MacIntyre bottom grab. The water was carefully drained from the grab sampler before sediment samples were taken. A field sampling manual was compiled by Lauenstein and Young (1986) to ensure uniformity for sediment and fish sample collection. The collection process is outlined in Figure I.1.

##### 3.1.2.1.1. Organic sample collection

Surface skims taken from the top 3 cm of three separate box cores or grab samples were frozen in the field, and composited in the laboratory to yield one sediment sample per station (Figure I.2). Composites contain approximately equal weights of material from each of the three skims. This procedure was performed at three stations per site.

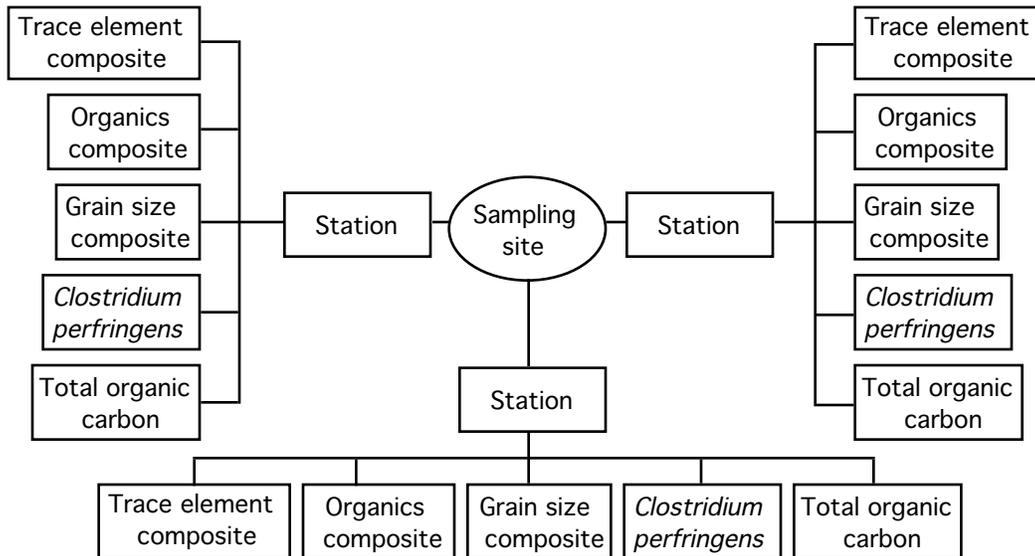


Figure I.1. Schematic of sediment collection procedure.

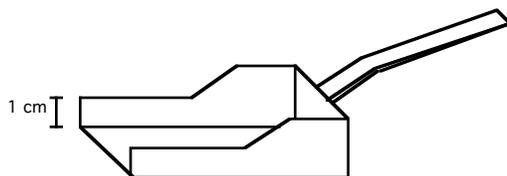
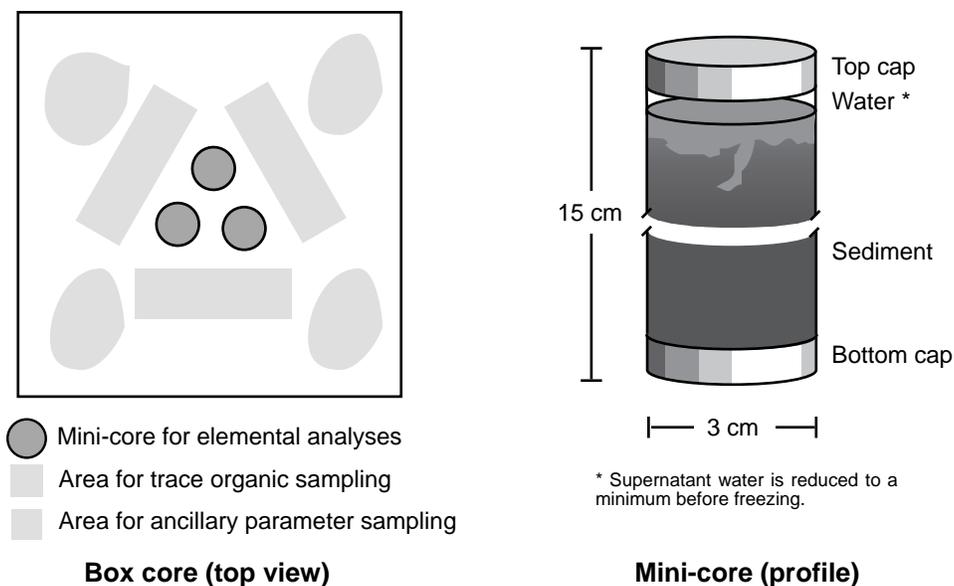


Figure I.2. Schematic of box core and mini-core used by the NBSP, and the sediment scoop used by the MWP to collect surficial sediments.

### 3.1.2.1.2. Major and trace element sample collection

Three 3 x 15-cm mini-cores were collected from each of three box cores or grab samples at each station and frozen (Figure I.2). The mini-core samplers were acid-rinsed, plastic tubes made of cellulose acetate butyrate. The mini-cores were kept upright so as not to disturb the surface sediment. Sediment samples were obtained from the central area of the box core or grab sample to avoid sample contamination from the walls of the sampling device, and to avoid collection of older sediments that may have been exposed as the sampler moved down through the sediment. If necessary, excess supernatant water was allowed to flow out by extruding the sediment to the top of the plastic tube of the mini-core prior to freezing.

Any excess overlying water was drained from the frozen mini-cores by piercing the lower end caps and allowing the mini-cores to thaw in a vertical position; suspended material in the water settles onto the surface of the sediment as the water drains. The top 3 cm of each drained mini-core was extruded in the laboratory, and the remaining sediment was discarded. Sediments from each minicore were dried separately and homogenized before compositing using equal weights of material from each of the mini-core samples. As with organic sample collection, this procedure was performed at three stations per site.

### 3.1.2.2. Packaging

Sediments collected for organic analyses were placed in glass jars with Teflon lid liners. Sediments collected for trace element analyses were retained in their plastic tubes. All sediments were frozen aboard ship. Since 1990, laboratory samples have been maintained in ultra-cold freezers (-80°C) until analyses were performed.

### 3.1.3. Tissue

During the first years of the NBSP, two technical memoranda were prepared detailing methods for the sampling of fish tissues (Lauenstein and Young 1986; and Lauenstein *et al.*, 1987). These methods were developed to minimize introduction of contaminants during sample collection or specimen dissection. The possibility of contamination being introduced at time of sample collection is discussed by Patterson and Settle (1976).

#### 3.1.3.1. Collection

Fish were primarily collected with Otter trawls towed by NOAA research vessels or their associated boats. Occasionally, along the Southeast and Gulf Coasts, fish were taken with hook and line, or with gill nets. These alternate collection methods were necessary because larger fish, such as older Atlantic croaker, were able to avoid an Otter trawl, or were found in untrawlable habitats such as shallow water, along marsh edges, and over oyster reefs.

Fish in the correct size range were dissected in the onboard laboratory immediately after collection. This ensured that a determination could be made regarding whether sufficient material had been collected and whether the sample material was of high quality. If either one of these criteria was not met, the opportunity existed to continue sample collection. Also, field dissection minimizes contamination problems associated with dissection of frozen fish samples. Frozen fish tissues, when thawed, may lose their integrity and one tissue type may contaminate another (e.g., a liver sample could be contaminated by PAH metabolites from the bile duct). Fish tissues for histopathological examination must be prepared in the field because freezing will destroy the morphology of tissue (C. Stehr, NOAA/NMFS/NWFSC, Seattle, WA, personal communication, 1993).

#### 3.1.3.2. Fish dissection environment preparation

Because most fish were dissected onboard ship, a special effort was made to develop an environment as close to clean-room conditions as possible. All fish were dissected in positive pressure laminar flow hoods. Air was drawn into the laminar flow hood from above and filtered by a high efficiency particle attenuator (HEPA) filter before it passed over the fish samples.

Stainless steel tools were used to dissect fish for organic analysis. Titanium tools were used to dissect fish for trace metal analyses because tools made of this element do not pose the problem of introducing nickel, chromium, and/or iron into the specimens to be analyzed. Specimens were analyzed for the latter three elements by the NS&T Program. After knives had been sharpened, and before dissections began at a new site or of a new species, the dissection equipment was thoroughly cleaned with detergent solution, rinsed extensively with tap water, rinsed in distilled or high-purity water (i.e., milli-Q or HPLC-grade water), rinsed with isopropanol<sup>Δ</sup> under a fume hood, followed by a rinse with distilled water, and placed on a similarly cleaned Teflon cutting board that was allowed to air-dry in the laminar-flow hood. Between individual fish of the same species at the same site, the tools were rinsed with

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<sup>Δ</sup> Before 1990, dichloromethane was used instead of isopropanol.

distilled water before any fluid or tissue had a chance to dry on the knife (C. Stehr, NOAA/NMFS/NWFSC, Seattle, WA, personal communication, 1993).

#### 3.1.3.3. Fish specimen preparation

All fish of the desired species were measured and individual total standard and/or pre-anal lengths recorded. Fish lengths were then plotted to prepare a histogram, frequently resulting in a normal distribution. The minimum accepted length varied among species, but was that of a sexually mature fish (Stehr *et al.*, this document). While a number of different species of benthic fish were collected, the general size criteria were: flatfishes of less than 15 cm total length were not retained, and no roundfishes of less than 12.5 cm total length were retained for dissection and subsequent analysis.

Fish were dispatched using a filleting knife or scalpel to sever the spinal cord just posterior to the brain. The fish were then wiped with a paper cloth to remove as much mucus as was practical.

#### 3.1.3.4. Onboard laboratory requirements for fish dissections

Three sets of sampling tools were used to remove fish tissues for analysis. One set was used to cut through the body wall or make the initial cuts through the epidermis for fish muscle dissection. A second set was used to collect the liver and other internal tissues that were analyzed for organic contaminants. The third set, consisting of a Teflon knife and polyamide forceps, was used for collecting liver tissue for trace element analyses. The second set of cutting instruments avoids chemistry samples from being contaminated with dirt and mucus from the surface to the fish, while the third set of tools avoids introducing trace element contamination. Dissection sequences are shown in Figure I.3.

#### 3.1.3.5. Tissue dissection

The fish body cavity was opened with a scalpel or scissors and the gender recorded. Dissection tools were cleaned prior to use on the next fish.

Using surgical scissors from the set of tools used only on internal tissues, the liver and gall bladder were cut from the surrounding tissues and removed from the body cavity. Care was taken so that bile did not spill on the liver during this step. The liver was then divided, in the order shown (Figure I.4), among the five different types of analyses to be performed: trace elements, histopathology, organic contaminants, AHH, and DNA adducts.

Stomach contents were collected to determine fish prey organisms and to quantify the concentration of organic contaminants in prey organisms.

Fish muscle tissue was removed (Figure I.5) using the following procedure.\* The fish was placed with the eyed or left side facing up. A series of four cuts were made to expose a rectangular subsection of muscle. The scalpel was wiped and rinsed with distilled water between cuts to remove scales and as much mucus as possible. The first cut was 100 to 150 mm long and extended from behind the head parallel to and about 5 to 10 mm dorsal to the lateral line. The next cut was above and parallel to the first, just below the fin ridge. Then two perpendicular cuts were made at the ends of the parallel cuts to complete a rectangular incision.

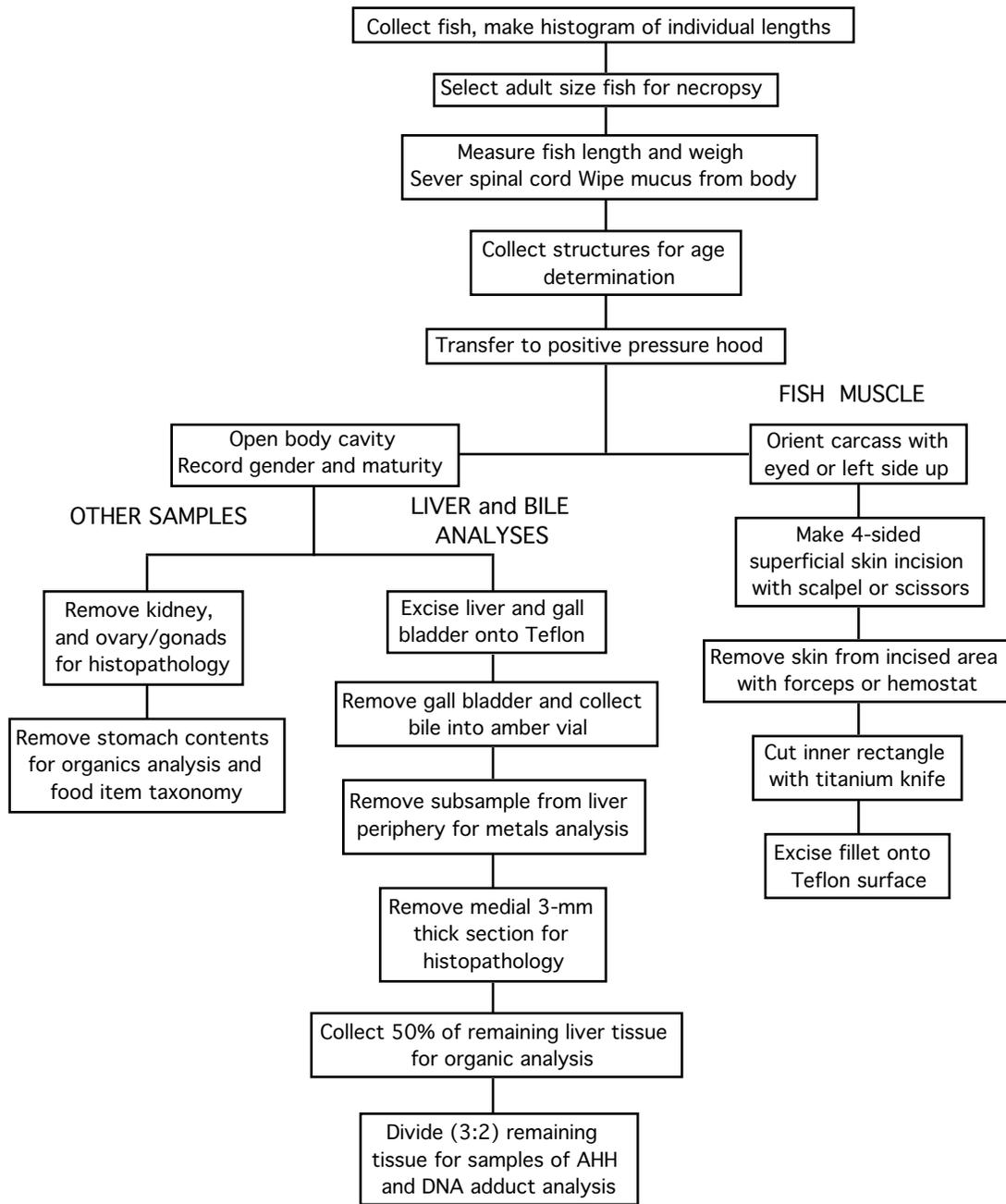


Figure I.3. National Benthic Surveillance Project fish tissue necropsy protocol.

The scalpel was used to lift the edge of the skin along the cut line at the posterior end of the rectangular cut. The fish tail was held with one hand and the edge of the skin was pulled back using forceps or a hemostat held in the other hand. The skin was pulled back from the rectangular cut to expose the muscle tissue mass. A layer of adipose tissue lies along the dorsal fin ridge. This tissue was not to be taken with the muscle tissue subsample because it is more fatty than the other muscle tissue and may contain more organic contaminants than the rest of the muscle tissues.

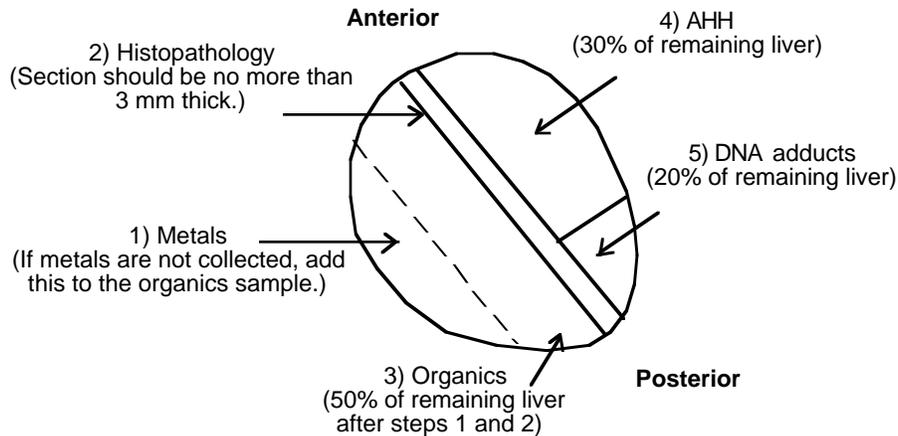


Figure I.4. Analytical destination of dissected liver tissues (Stehr *et al.*, this document). Percentages of liver to be taken apply to the remaining liver after sub-samples for metal analysis and histopathology have been taken.

The "core" of the muscle tissue mass within the rectangular cut was cut free and removed with a titanium knife. Extreme care was taken to assure that neither the contaminated rectangular cut line nor the fish exterior was contacted either by the titanium knife or by the cored muscle sample. Polyamide forceps were used to transfer muscle tissues to storage containers for samples to be analyzed for trace elements. Contaminants are found in lower concentrations in fish muscle tissues than in other tissues, such as liver, therefore prevention of sample contamination is extremely important.

#### 3.1.3.6. Packaging

Fish analytical samples were stored according to their source and their use. Flatfish otoliths used for age determination were stored dry in a test tube and roundfish otoliths were stored in 70% ethanol. Fish muscle tissue was placed in Teflon bags, sealed, and frozen. On occasion, whole juvenile fish were frozen for later dissection and trace element analysis. Fish liver tissues were divided during the dissection process between those to be characterized for contaminants and those to be studied for histopathology. Liver histopathology samples were placed in tissue cassettes in Dietrich's fixative.\* When gross liver pathology was noted on liver tissues, histology samples were also taken from the heart, upper intestine and spleen, placed in tissue cassettes and preserved in Dietrich's fixative. Bile was stored in amber vials, to reduce possible photo-oxidation, and frozen. Liver tissue used for quantifying trace elements were placed in acid-rinsed plastic vials and frozen. Liver tissues to be analyzed for trace organic contaminants were placed in dichloromethane-rinsed scintillation vials with Teflon-lined lids and frozen. Liver tissues used to quantify aryl hydrocarbon hydroxylase and xenobiotic-DNA adducts were stored in cryovials and immediately frozen in liquid nitrogen. Stomach contents were placed in dichloromethane-rinsed glass jars capped with Teflon-lined lids and frozen (Stehr *et al.*, Volume II, this document).

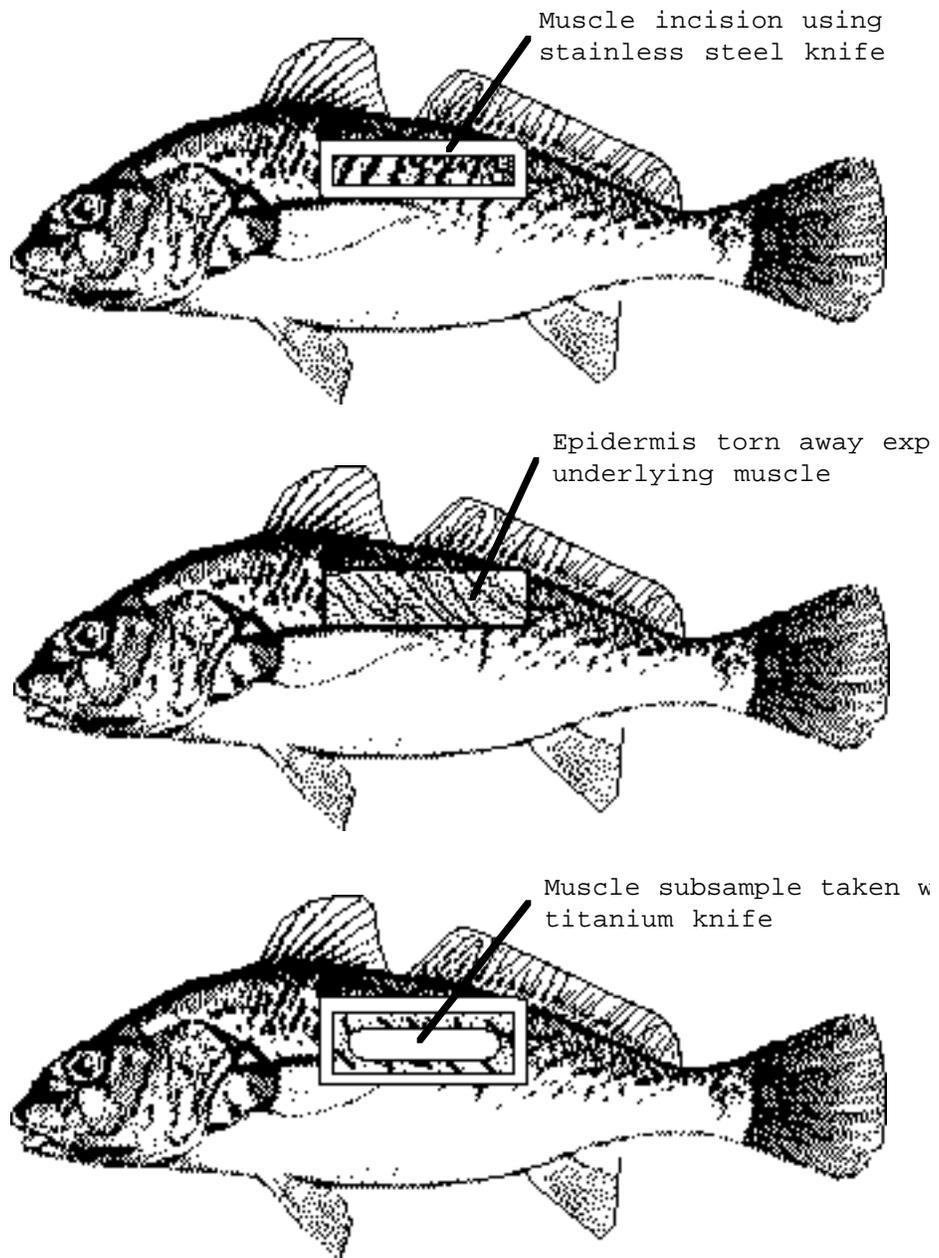


Figure I.5. Three-step fish muscle dissection sequence [Atlantic croaker (*Micropogonias undulatus*) shown].

### 3.2. Mussel Watch

NOAA's Mussel Watch Project began in 1986 and has collected bivalve mollusks on an annual basis since that time. Sediments were also collected and contaminants quantified for all sites during the first two years of the project and sediments have been collected and quantified at new sites when they were established. Originally, in 1986, samples were collected and analyzed from 150 sites around the United States, including Alaska and Hawaii. By 1992, the project had sampled over 250 sites and included sites in the Great Lakes and Puerto Rico. Field collections and laboratory work have been performed primarily by non-NOAA contract laboratories.

Since 1986 Texas A&M University has been responsible for Gulf Coast sample collection and analyses, while Battelle Ocean Sciences has performed the collection and analytical work for the East Coast. Collection and analyses of samples from California and Hawaii, with minor exceptions (Table I.8), was performed by SAIC from 1986 through 1989. For those years, Battelle Ocean Sciences was responsible for the Oregon, Washington, and Alaska portions of the West Coast work. Beginning in 1990, Battelle has been responsible for the entire West Coast as well as the East Coast work. Sites in the Caribbean are collected and analyzed by TAMU. Samples from the Great Lakes have been collected by NOAA and analyzed by Battelle.

Sampling for the MWP for all years occurs between mid-November and the end of March. While the entire sampling effort occurs during this time frame, the criterion for the annual sampling of a MWP site, once established, requires that samples be collected within three weeks of the date the site was first sampled. The intention of sampling all sites in this time frame is to avoid the possible effects of spawning on chemical concentrations (Phillips, 1980).

#### 3.2.1. Site designation

Mussel Watch Project sites carry both location and site names. The location name refers to a general geographic area or estuary (e.g., Boston Harbor). The site name defines a more specific location within the designated geographic area (e.g., Boston Harbor, Deer Island). Each site has a unique four letter MWP site code. Usually, the first two letters refer to the site location, while the second two letters refer to the exact site (e.g., BHDI for Boston Harbor, Deer Island). Site acronyms can be differentiated between the two programs because those of the NBSP contain five letters, while those of the MWP contain four. For more information about site locations see Table A.48 (Appendix A), and Lauenstein *et al.* (1993). In most cases, mollusk populations are stable and may be sampled year after year. At each site, three bivalve mollusk and three sediment stations were sampled. Sediment collections are made as close to the bivalve collection locations as possible.

Site latitudes and longitudes are certain to within 20 m. This level of accuracy is possible, in part, because by 1992 most MWP sites were defined using Global Positioning System technology. Prior to 1992, for the East and West Coast sites, and prior to 1990 for the Gulf coast sites, the primary method used to return to site locations was with Loran-C. These site location determinations were augmented by using triangulation to significant landmarks while in the field. Sites were also extensively photographed, including the use of aerial photography for sites along the Gulf of Mexico. Latitudes and longitudes (Tables A.47 and A.48, Appendix A) were derived from both GPS information, and from plotting sites locations on NOAA charts.

Table I.8. Laboratories analyzing Mussel Watch Project samples.

	Year			
	1986-1987	1988	1989	1990-1992
Trace Elements				
East Coast	Battelle	Battelle	Battelle	Battelle
Gulf Coast	TAMU	TAMU	TAMU	TAMU
West Coast				
California	SAIC	SAIC <sup>Δ</sup> SAIC <sup>◇</sup>	Battelle	
Oregon	Battelle	Battelle	Battelle	Battelle
Washington	Battelle	Battelle	Battelle	Battelle
Alaska	Battelle	Battelle	NS	Battelle
Hawaii	SAIC	SAIC NS	Battelle	
Great Lakes			Battelle*	
Organic Contaminants				
East Coast	Battelle	Battelle	Battelle	Battelle
Gulf Coast	TAMU	TAMU TAMU	TAMU	
West Coast				
California	SAIC	SAIC SAIC	Battelle	
Oregon	Battelle	Battelle	Battelle	Battelle
Washington	Battelle	Battelle	Battelle	Battelle
Alaska	Battelle	Battelle	NS	Battelle
Hawaii	SAIC	SAIC NS	Battelle	
Great Lakes			Battelle*	

Battelle - Battelle Ocean Sciences, Duxbury, MA, and Sequim, WA.

TAMU - Geochemical and Environmental Research Group of Texas A&M University, College Station, TX.

SAIC - Science Applications International Corporation, Inc.

<sup>Δ</sup> Sn analyzed by Battelle.

<sup>◇</sup> Se and Sn analyzed by Battelle.

NS - Not sampled

\* Great Lakes sites were first sampled in 1992.

### 3.2.2. Sediments

When taken, sediment samples are collected concurrently with bivalve samples. The same rationale is applicable for collecting sediments in the MWP as for the NBSP. Sediments are the matrix that allow the greatest possible spatial comparisons to be made across sites and between monitoring projects.

Samples were taken from the top centimeter of sediments collected for the MWP, while the first three centimeters were taken for analyses by the NBSP laboratories. The intent of collecting surficial sediments is to quantify "recent" contaminant inputs. The difference between sediment collection depths of the two monitoring projects might be expected to lead to different results. In an undisturbed environment, sediments collected closer to the surface would be considered to be more recent than those samples collected at a greater depth. Sediments are frequently disturbed by benthic fish, benthic invertebrates, and physical phenomena such as wave action and storm surge.

Sediment site selection criteria are as follows.

- The site shall be subtidal (never exposed at lowest low tides), and should be a low energy depositional environment as evidenced by surficial sediment containing at least 20% fine-grained material ( $\leq 64$  microns) on a dry weight basis.
- The site shall be exposed to the same water mass as the corresponding bivalve site.
- The site should be located as near as possible to, and preferably not more than 2 km from, the bivalve site.
- The site shall integrate contaminants from multiple sources in the surrounding area, but not reflect inputs from an individual point source of contamination.

The procedure followed to establish a sediment sampling site is outlined in Figure I.6. These criteria were established during 1987, the second year of the project.

Since sediment collection methods differ slightly between Battelle and TAMU, they are discussed separately.

#### 3.2.2.1. Collection

##### 3.2.2.1.1. East and West Coasts

East and West Coast sediment samples were obtained by Battelle using a specially constructed Kynar-coated Young-modified Van Veen bottom grab (Battelle, 1987a). The grab sampler was cleaned after each use and between each site. The sediment scoop (Figure 1.2) was cleaned between each grab sample by washing with soap and water, rinsing with distilled water, rinsing with methanol or acetone, and rinsing with dichloromethane. Solvent rinses are collected in specially designated waste containers and returned to the laboratory.

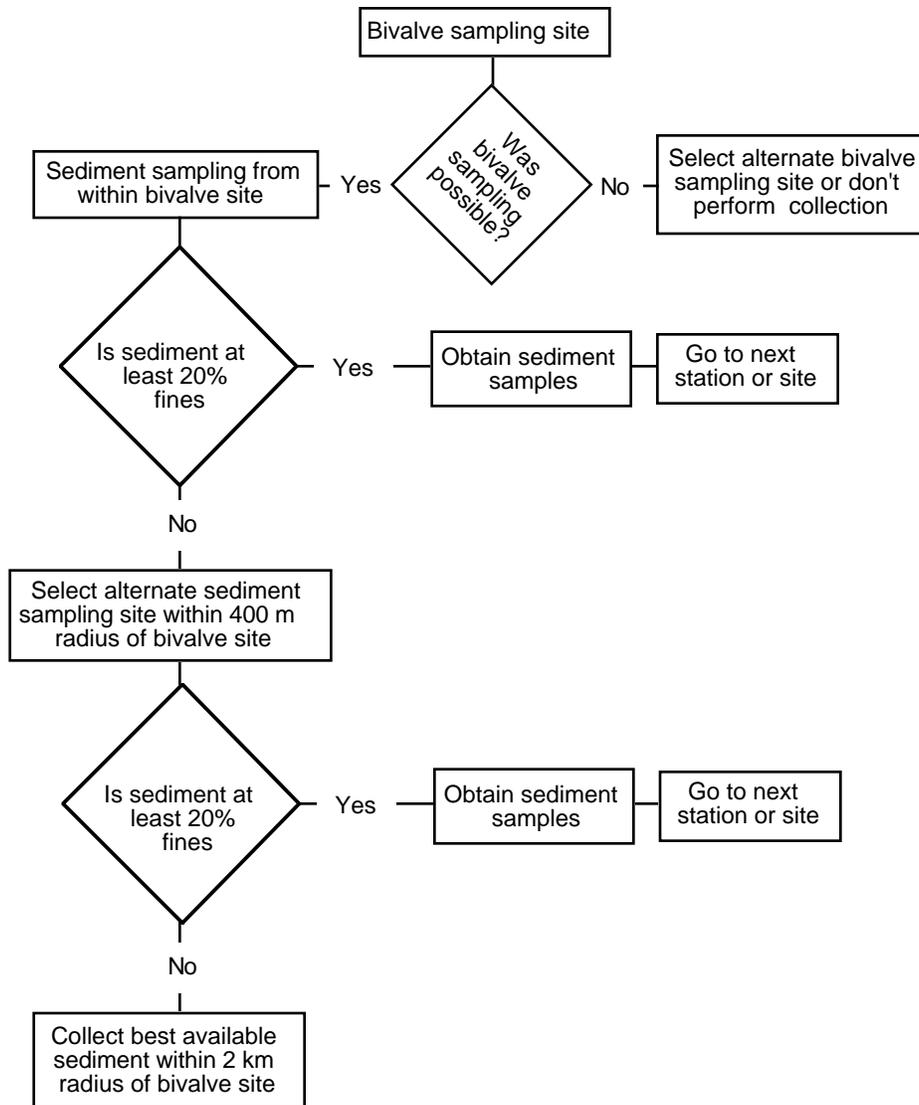


Figure I.6. Sediment site selection criteria (Battelle, 1987a).

Surface sediment skims taken from the top 1 cm of three separate grab samples were composited in the field to yield one sediment sample per station (Figure I.7). Sediments were taken with a hand held scoop that was calibrated to take the top centimeter of sediment. The scoop was coated with Kynar [poly(vinylidene fluoride)] so that trace elements from the scoop did not contaminate the sediments being sampled. The order of operations followed were:

Overlying water was drained from the grab using a Teflon siphoning tube so that the sediment surface was not disturbed.

The top 1 cm of sediment was taken using the Kynar-coated sediment scoop. Sediment adjacent to the grab walls was avoided.

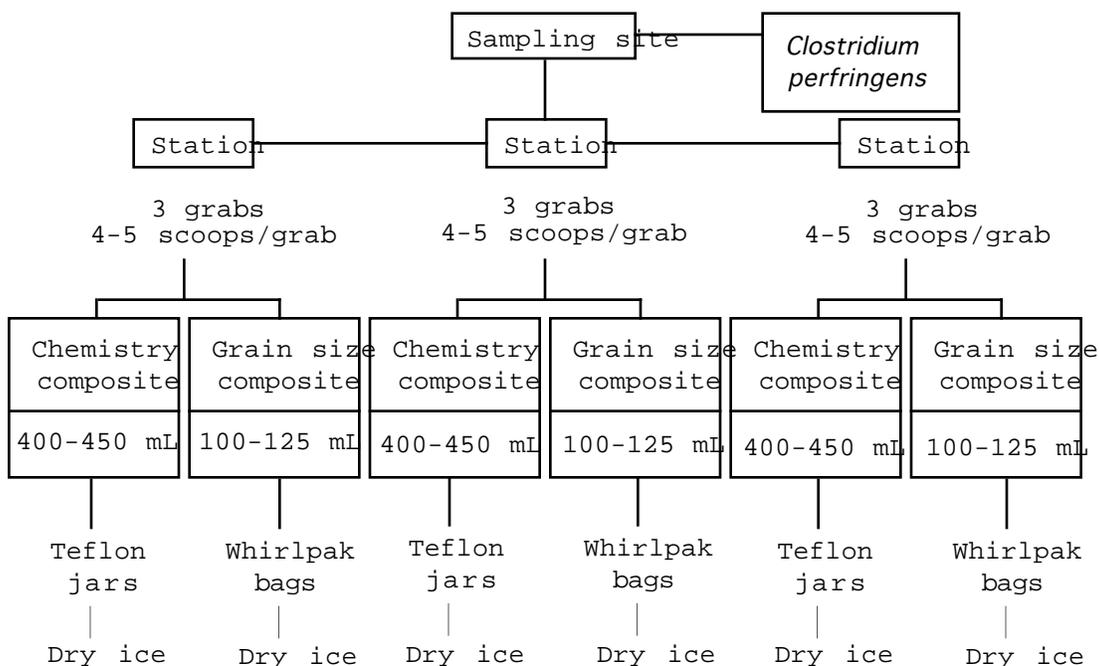


Figure I.7. East and West Coast sediment field sampling.

Sediment samples were placed into separate station containers: 500-mL pre-cleaned Teflon jar for sediment chemistry, Whirlpak bag for grain size, and sterile specimen cup for *Clostridium perfringens* sediment samples.

Samples were transferred to a cooler containing dry ice and returned to the laboratory at the end of each day (Battelle, 1989).

Station composites were represented by approximately equal weights of material from each of the three grabs.

### 3.2.2.1.2. Gulf Coast

Field sampling methods used along the Gulf Coast are summarized in Brooks *et al.*, 1988. Gulf Coast sediments were collected by a stainless steel box core or a hand held Teflon-coated sampling scoop. The hand held scoop was used for the primary sediment collection at only a few sites along the Gulf Coast, where depositional sediments were found in shallow, low energy environments, i.e., low tidal range and current flow. Prior to each use, the scoop was cleaned and rinsed with acetone and dichloromethane was used to remove traces of residual organics. Waste solvents were collected and returned to the laboratory for disposal.

Surface skims were taken from the top 1 cm of three separate box corer samples and were composited in the field to yield one sediment sample per station (Figure I.8). Station composites were represented by approximately equal weights of material from each of the three skims. Organic samples were stored in either Teflon or glass jars with aluminum foil lid liners. Sediments quantified for major and trace elements were stored in either Teflon jars or Ziploc bags. Samples were frozen in the field for shipment back to the laboratory.

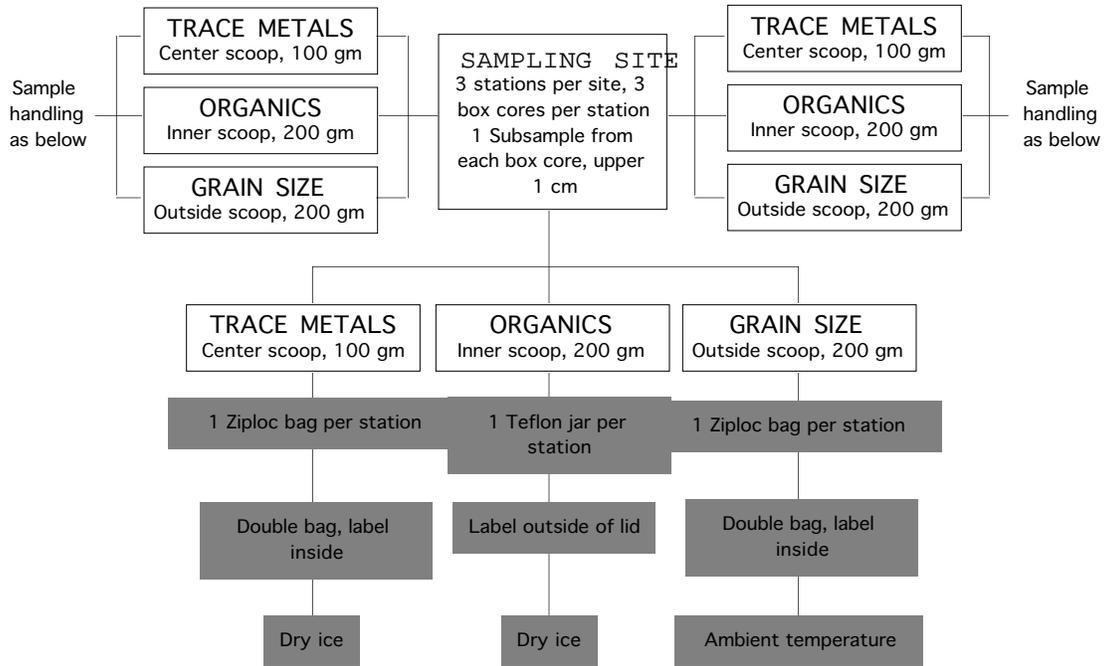


Figure I.8. Gulf Coast sediment field sampling (Brooks *et al.*, 1988).

### 3.2.2.2. Packaging

Battelle prepared sediment and mollusk samples for overnight shipment back to the laboratories at the end of each day's field collection. Samples designated for contaminant analyses were shipped back to the laboratories on dry ice. Samples to be used to quantify mollusk gonadal index were preserved in Dietrich's solution.

Samples were handled differently by TAMU. Until 1991, TAMU brought a mobile laboratory into the field that was transported from site to site by truck. This mobile laboratory allowed field scientists to prepare samples in the field. Sediments collected for grain size analysis were stored at ambient temperatures or refrigerated, but not frozen, in Whirlpak and/or Ziploc bags. Sediment ancillary measurements, such as total organic and inorganic carbon, were performed on aliquots of the samples taken for organic analyses; thus, no additional samples or processing was required in the field. Since 1991, TAMU has sent all samples directly back to the laboratory, on dry ice, with no special preparation or characterization of samples occurring in the field.

### 3.2.3. Tissues

An offshore subtidal bivalve collection site is defined as being a circle with a 400 m radius around the site center. Intertidal shoreline sites are defined as being 100 m in length along the shore or breakwater. Sediments may be collected within 2 km of the bivalve site center if depositional sediments can not be found closer to the intertidal sites or within the 400 m radius of the bivalve site (Battelle, 1987a). For certain West Coast sites where depositional (i.e., fine grained) sediments could not be found associated the bivalve site, sediment and bivalve collections were made even further apart than the 2 km criterion. In these cases, the sediment site received a different site acronym from that of the bivalve site.

Bivalve collection criteria established for the MWP are:

- Indigenous populations of mollusks must exist at a potential sampling site since the monitoring effort does not use caged mussels.
- The NS&T bivalve sites coincide with historical monitoring sites (i.e., EPA Mussel Watch monitoring sites) when feasible and when all other criteria are met.
- The site contains indigenous bivalves of a suitable size (5 - 8 cm for mussels, 7 - 10 cm for oysters) available for collection.
- The NS&T Program is not intended to quantify contaminants in hot spots. Rather, mollusk collection sites are selected to be representative of the body of water sampled. Therefore, Mussel Watch sites are not knowingly located near waste discharge points or in poorly-flushed industrial waterways.
- Sample substrates are limited to natural substrates or structures made of natural materials such as rock (including rip-rap and jetties), sand, or mud. Collection of samples on buoys and preserved wooden structures can yield artificially high results for some contaminants being quantified by the NS&T Program.
- The site is suitable for follow-up sampling (i.e., it is not anticipated that the site will be physically disrupted by development or that the mollusk population depleted by sampling).

The number of different molluscan species is kept to a minimum in order to make the greatest spatial data comparisons possible. Blue mussels (*Mytilus edulis*) are collected on the East Coast at sites from Maine to Delaware Bay. When MWP collections began in 1986, two West Coast mussel species were identified: *Mytilus edulis*, usually collected at inshore sites, and *Mytilus californianus*, usually sampled at open coastal sites. From Delaware Bay south and throughout the Gulf of Mexico, the American oyster (*Crassostrea virginica*) was sampled. A separate species, *Ostrea sandvicensis*, is sampled at the Hawaiian Islands sites. In order to sample mollusks in the Florida Keys, the addition of a new species was necessary, the Smooth-Edge Jewel Box (*Chama sinuosa*). During the seventh year of the Project, samples were collected in Puerto Rico. The sites in Puerto Rico supplied the species *Crassostrea rhizophorae*, a species commonly known as the Mangrove Oyster and closely related to *C. virginica*. Zebra mussels (*Dreissena polymorpha*) have been collected in Great Lake sites since 1992.

Within North American waters, several different genetic stocks have recently been identified for the *M. edulis* species complex. *M. galloprovincialis*, common in the Mediterranean, has been reported to be the dominant California member of the Family Mytilidae, other than *M. californianus*, (McDonald and Koehn, 1988). Washington and Oregon mussels are thought to be predominantly the species *M. trossulus* (McDonald and Koehn, 1988). The northeast sites identified as supplying *M. edulis* probably do, but *M. trossulus* is found further north in Canadian waters. It is unclear, however, whether or not allozymes and morphological characteristics are sufficiently different to define these organisms as separate species. A number of authors have referred to these as independent species (Seed, 1978; Skibinski *et al.*, 1980 and 1983; Gosling, 1984; McDonald and Koehn, 1988). Seed (1992) concludes that despite the lack of any absolute reproductive barrier and the massive potential for dispersal, populations of *M. edulis*, *M. galloprovincialis*, and *M. trossulus* comprise relatively homogeneous groups, each maintaining a unique genetic and morphological phenotype across vast distances. It should be noted that the purpose of a monitoring program is to quantify the spatial extent and temporal change of environmental contamination so whether or not contaminants in separate species are quantified is not so much of concern as is whether or not contaminant levels are comparable

between the organisms studied. If the ability of organisms to concentrate environmental contaminants are the same, data comparisons are valid regardless of species.

Data were compared between two species groups, *M. edulis*<sup>Δ</sup> vs. *M. californianus* and *M. edulis* vs. *C. virginica* (NOAA, 1989; O'Connor, 1990). Comparisons between *M. edulis* and *C. virginica* resulted in no statistically significant differences between their organic contaminant concentrations, while there were statistically significant differences for certain trace elements (Figure I.9). Oysters clearly have a greater affinity for silver, copper, and zinc than do mussels, while mussels have a greater affinity for chromium and lead. There is no discernable difference in the ability of the two *Mytilus* species to concentrate organic or trace element contaminants. With data for species as divergent as *M. edulis* and *M. californianus* able to be compared, it is assumed that the small differences between *M. edulis* and its hybrid/congeners make no difference from a monitoring stand point.

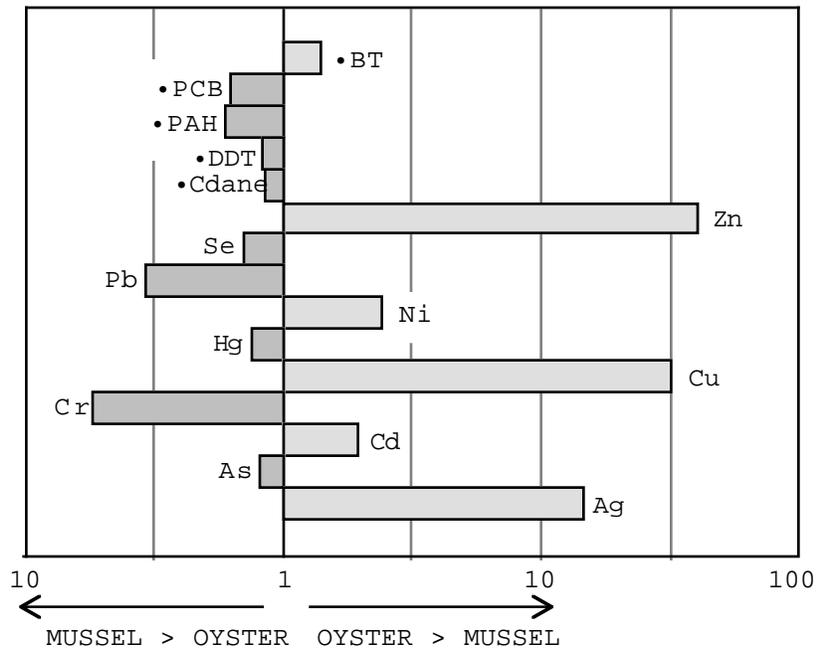


Figure I.9. Factors by which mean contaminant concentrations in oysters (*C. virginica*) differ from those in mussels (*M. edulis*) collected at the NOAA NS&T Housatonic River site in Long Island Sound in 1989 ( $\Sigma$ BT is the sum of the concentrations of tributyltin and its breakdown products, dibutyltin and monobutyltin.  $\Sigma$ PCB is total PCB concentration calculated from the sum of the concentrations of the 18 congeners determined as part of the NS&T Program.  $\Sigma$ PAH is the sum of the concentrations of 24 PAHs.  $\Sigma$ DDT is the sum of the concentrations of DDT and its metabolites.  $\Sigma$ Cdane is the sum of the concentrations of the two major constituents of chlordane mixtures, *cis*-chlordane and *trans*-nonachlor, and of those of two minor constituents, heptachlor and heptachlor epoxide.) (Redrawn from O'Connor, 1992).

### 3.2.3.1. Bivalve mollusk collections

<sup>Δ</sup> *M. trossulus* may actually have been the species analyzed.

### 3.2.3.1.1. East and West Coasts

Information summarized below was derived from Battelle (1987a). The primary objective in sampling bivalves was to obtain three discrete composite samples from three stations (Figure I.10). When this division of the site into stations was not possible, a pool of bivalves representative of the site was collected and divided into 3 separate collections. Each collection was treated as if it were from a station. This latter procedure was usually necessary when the collection was made using a dredge, under other subtidal collecting conditions, or when the bivalve population was concentrated in a very small area.

A station-sample consisted of 150 to 300 mollusks, depending on size and species. These were divided into groups as described in Figure 10. For both mussels and oysters, 10 individuals per station were analyzed from 1986 to 1989 to determine the population's gonadal index. For 1990 and 1991, five organisms were characterized per station.

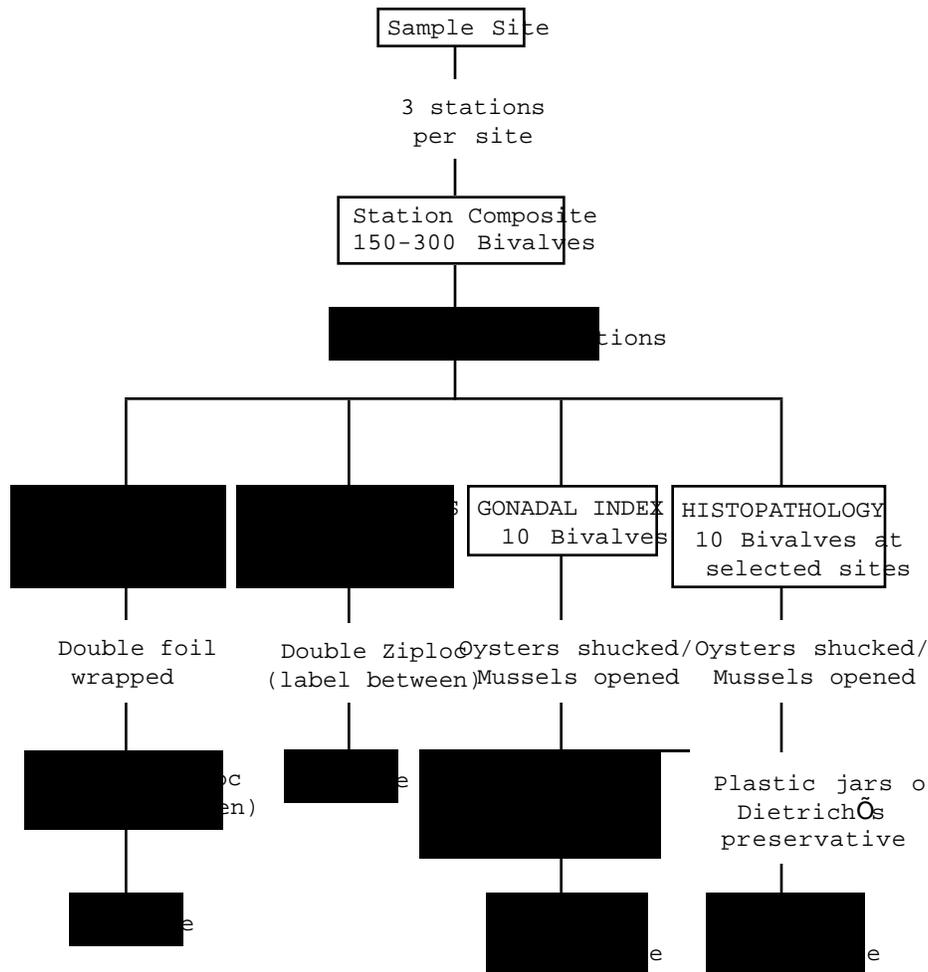


Figure I.10. East and West Coast bivalve mollusk field sampling (Battelle, 1987a).

Field teams collecting samples along the East and West Coasts employed several different collecting techniques depending upon the station depth, species, and environmental conditions at the site. Collection techniques were also constrained by sampling permits issued to field teams.

A bivalve dredge was used in water deeper than 2 m. The dredge is a toothed skip dredge of stainless steel. The dredgebag is constructed of polypropylene mesh to minimize trace metal contamination from the steel chain used on other dredges of this type. Dredge and bag weigh approximately 75 lbs.

In water 2 to 2.5 meters deep where the bottom was relatively soft, bivalves were on occasion sampled with stainless steel tongs. Tong heads, toothed baskets generally 18 to 20 in wide, were dug into the bottom with a down-jabbing motion. The bivalves were brought to the surface by squeezing and lifting the tong handles.

In water less than 1 m deep, bivalves were also collected using a stainless steel pitch fork or quahog rake.

At some shoreline sites, the intertidal bivalve populations were also collected from natural substrate by hand. The field team wore polyethylene, rubber, or other non-contaminating gloves when removing the bivalves from the substrate. Bivalves were separated when found adhering to each other, and scrubbed with a nylon or natural fiber brush to remove adhering detritus.

#### 3.2.3.1.2. Gulf Coast

The following information is summarized from Brooks *et al.* (1988). Oysters were collected by hand, with tongs, or using a dredge. Where possible, hand collection was the method of choice. Intertidal and shallow subtidal sites were collected by hand. Loose oysters were simply picked from the reef and separated from attached shell debris or other oysters using an oyster knife or chipping hammer. In some areas, the use of a dredge was prohibited (e.g., Appalachicola Bay). Subtidal sites in these areas were sampled using oyster tongs, and the oysters separated from one another using oyster knives or chipping hammers. At the deeper subtidal sites and where oysters were obtained directly from commercial oyster fishermen from privately leased sites, the oysters were collected with steel oyster dredges. Clumps of oysters and shell were separated into individual oysters.

#### 3.2.3.1.3. Field collection changes

The MWP collection and analytical techniques were modified in the seventh year (1992) of the project. Bivalve site selection criteria and collection techniques remained the same, but the number of specimens collected at each station was reduced to that necessary for one composite sample per site. This one composite contains material from three stations. Three stations were composited per site such that site averages from previous years, for elements and organic contaminants, could still be statistically compared to the newer site data. The environmental variability found between the earlier three station per site analyses are now incorporated into one site composite analysis.

### 3.2.3.2. Packaging

#### 3.2.3.2.1. East and West Coasts

##### 3.2.3.2.1.1. Organic samples

Sampling teams wearing polyethylene gloves: 1) double wrapped 30 clean mussels or 20 oysters in aluminum foil, 2) labeled foil appropriately and covered labels with clear packing tape, 3) placed foil-wrapped samples inside a Ziploc bag and sealed the bag, and 4) transferred samples to a cooler containing dry ice (Battelle, 1989).

Because the high gloss side of aluminum foil has been treated with organic compounds, foil that is used to package specimens is first solvent rinsed in the laboratory. The field team places the dull side (untreated side) against the samples.

##### 3.2.3.2.1.2. Major and trace element samples

Sampling teams wearing polyethylene gloves: 1) placed 30 mussels or 20 oysters in a Ziploc bag and sealed the bag, 2) labeled bag appropriately and covered label with clear packing tape, 3) placed bag inside another Ziploc bag and sealed the second bag, and 4) transferred the sample to a cooler containing dry ice (Battelle, 1989).

#### 3.2.3.2.2. Gulf Coast

The following field collection methods are described in Brooks *et al.* (1990). Prior to 1992, once mollusks were collected they were segregated and labeled according to station and replicate. Samples were stored in ice chests until the day's sampling was complete. At that time, they were transferred to the mobile laboratory for processing.

Oysters were scrubbed free of mud and debris using pure bristle brushes and water from the collection site. Oysters for organic and histopathological analyses were measured for length and displacement volume. A complete cross section of gonadal tissue was excised from each oyster and placed into an individual tube of Bouin's solution. A small snip of mantle tissue was also removed and placed into individual tubes of thioglycollate medium for the determination of the incidence of the oyster parasite *Perkinsus marinus*.

The remaining oyster tissue, after histopathology samples had been taken, were used for organic analyses. Tissues were excised from the shell and placed into a pint mason jar which had been combusted in a muffle furnace to completely remove trace organics. The tare and gross weight of the jar and contents (20 oysters) were recorded along with the individual lengths of the oysters. A Teflon liner was placed under the jar seal, and the labeled sample jar, one for each of three stations in a site, was frozen in the laboratory deep freezer. The field processing procedures of oysters for organics and histopathology is shown in Figure I.11.

A separate 20 oysters from each of three stations within a site were washed as above and set aside in plastic trays for metal analyses. The oysters were collectively measured for their initial displacement volume in a calibrated tank containing water. Because of their sharp edges and the need to ensure they remained closed until processing, the oysters were individually wrapped in singlefold paper towels and bound with rubber bands before they were double bagged in plastic Ziploc bags, labeled and stored in the freezers. All opening and further processing of oysters, measurement of shell length and second displacement volume, was performed at the analytical laboratories in clean room conditions.

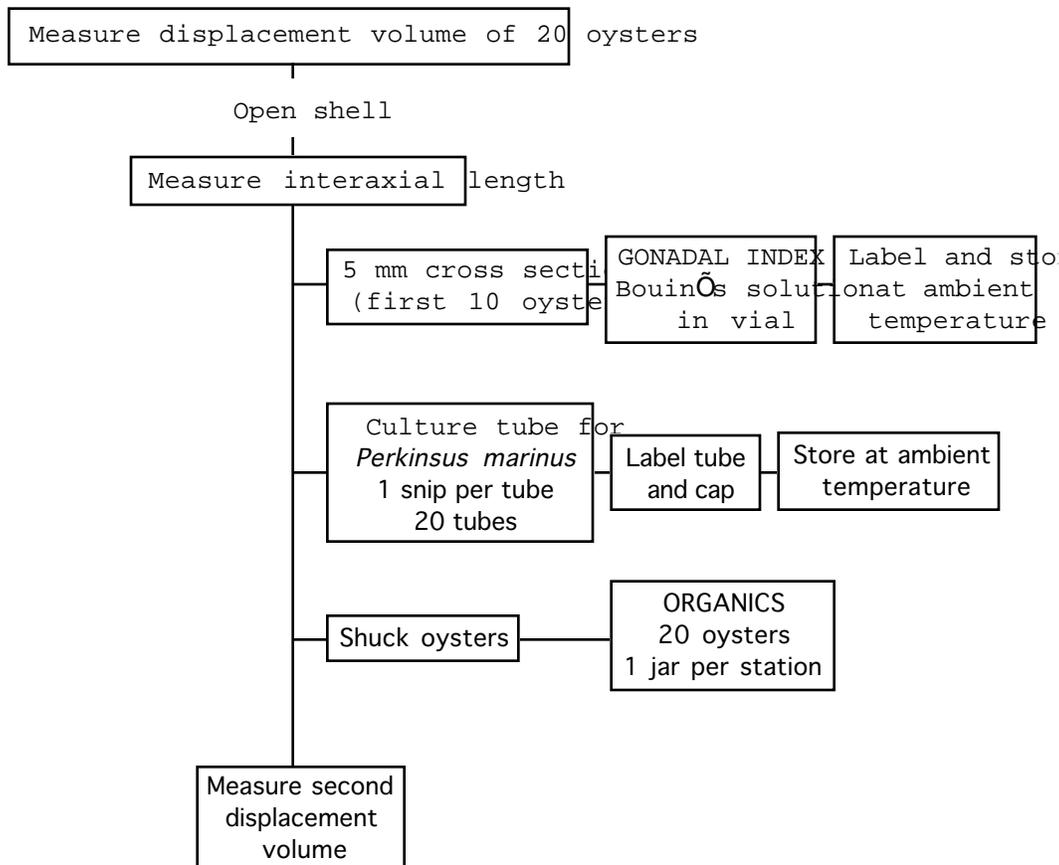


Figure I.11. Gulf Coast bivalve mollusk field sampling.

Frozen samples were stored in the mobile laboratory until they were returned to TAMU. Since 1991, TAMU has sent all samples directly back to the laboratory, on dry ice, with no special preparation or characterization of samples occurring in the field.

### 3.2.4. Ancillary measurements

In addition to the basic chemical and gonadal measurements, the MWP also performed ancillary measurements.

#### 3.2.4.1. Tidal horizon

Tidal horizon data were incorporated into the MWP to ensure that the field team collected intertidal specimens from the same population each year. Evidence exists that *M. galloprovincialis* is found higher intertidally than *M. edulis* (Gosling and McGrath, 1990). Sampling at the same tidal horizon also ensures that contaminant concentrations found in mollusks were not merely a function of the amount of time that mollusks were able to filter feed. For mussels, an increased concentration of cadmium was found with increased distance downshore (Roberts *et al.*, 1986). Stephenson *et al.* (1979) found statistically significant correlations between mussel tidal height and trace element concentrations for cadmium, chromium, aluminum, iron, and manganese.

#### 3.2.4.2. Depth

Depth to bottom is determined by depth sounder, weighted line, or, for shallow sites, a ruler. Depth is reported in increments of meters.

#### 3.2.4.3. *Perkinsus marinus*

Health of Gulf Coast oysters was quantified by the degree of infection by *Perkinsus marinus*, a protistan parasite. The degree to which oysters were infected by this protistan has been quantified for Gulf Coast oysters since 1986. In the mobile field laboratory, a small snip of mantle tissue is removed and placed into individual tubes of fluid thioglycollate medium (FTM) (Brooks *et al.*, 1989; Volume. II, this document).

#### 3.2.4.4. Shell size

Because a mollusk's body burden of metals is associated with individual organism size, individual shell size was determined for each specimen collected. Trace element concentration differences as a function of shell size, shell weight, and condition index have been noted by Phillips (1980), Fisher (1983), and Lobel *et al.* (1991b), respectively.

#### 3.2.4.5. Radionuclide samples

Radionuclide analyses were performed on mollusks collected from selected NS&T Mussel Watch Project sites in 1991 (Valette-Silver and Lauenstein, 1993). EPA's Mussel Watch Program of the 1970s also quantified radionuclides in addition to organic and inorganic contaminants. In 1991, the NS&T Program selected 36 MWP bivalve sites to be quantified. Bivalves to be analyzed (125 oysters or 200 mussels per site) were wrapped in aluminum foil and packaged in Ziploc bags, placed on dry ice, and shipped to TAMU. At the laboratory, mollusk samples were: 1) weighed wet, 2) placed in Mason jars and freeze dried, 3) weighed dry, and 4) sealed and shipped to the laboratory performing the radionuclide analyses.

#### 3.2.4.6. Coprostanol and *Clostridium perfringens*

Two ancillary parameters have been used to quantify the degree of mammalian fecal waste associated with marine sediments. During the first four years of the MWP, concentrations of coprostanol, a fecal sterol, was measured. In the fifth year, 1990, that chemical measurement was replaced with measures of *Clostridium perfringens*, a bacterium found in the mammalian intestinal tract. Equipment cleaning and sample collection are the same for *C. perfringens* analyses, as for grain size samples. At each of the three stations, approximately 100 mL of surface sediment from the top 1 cm was placed in a sterile sample container and frozen. Samples were not thawed and refrozen as this adversely affects the analysis (Battelle, 1990a; Emerson and Cabelli, 1982).

#### 3.2.4.7. Gonadal index

From 1986 through 1989, 10 bivalves were collected and analyzed from each station for the degree of gonadal maturation. Starting in 1990, 15 bivalve specimens were required for the gonadal index sample from each site. During the 1992 analytical year, analyses for gonadal state was not performed for each sampled site.

#### 3.2.4.8. Temperature

Water temperature was recorded at every site. Water temperature was measured to 0.1° C at each bivalve site. Measurements were made directly using a surface-deployed temperature probe. Additionally, temperature may have been measured from water samples collected with a Niskin bottle or equivalent, by using a portable digital thermometer or calibrated glass mercury thermometer. Before and after each phase of the field program, all thermometers were calibrated against a National Institute of Standards and Technology (NIST) calibrated mercury thermometer (Battelle, 1987a).

#### 3.2.4.9. Salinity

East and West Coast salinity was measured in parts per thousand (‰) with a refractometer at each bivalve site (Battelle, 1987a).

Gulf Coast salinity was measured either at the site with a temperature compensating refractometer or a small sample was returned to the mobile field laboratory where salinities from a number of collected sites were measured at constant temperature with a calibrated refractometer (Brooks *et al.*, 1990).

### 4. QUALITY ASSURANCE

The quality of the analytical data generated by the NS&T Program is overseen by the QA Project component, which has been in operation since 1985 and is designed to document sampling and analytical procedures, and to reduce intralaboratory and interlaboratory variation. The QA Project documentation will facilitate comparisons among different monitoring programs with similar QA activities and thus will extend the temporal and spatial scale of such programs. To document laboratory expertise, the QA Project requires all NS&T laboratories to participate in a continuing series of intercomparison exercises utilizing a variety of materials. The organic analytical intercomparison exercises are coordinated by the NIST, and the inorganic exercises by National Research Council (NRC) of Canada.

#### 4.1. Approach

##### 4.1.1. Methodology

NS&T does not specify analytical methodology. Laboratories can use any analytical procedure as long as the results of the intercomparison exercises are within certain specified limits of the consensus values. This allows for the use of new or improved analytical methods or instrumentation without compromising the quality of the data sets. It also encourages the contractor laboratories to use the most cost-effective methodology while generating data of documented quality.

##### 4.1.2. Standard reference and control materials

The analysis of reference materials, such as the NRC Certified Reference Materials (CRMs) and NIST Standard Reference Materials (SRMs), and of control materials generated for use by NS&T labs as part of the sample stream, is required. Analytical data from all control materials and all matrix reference materials are reported to the NS&T Program office. These data are stored in the NS&T Program office.

#### 4.1.3. Procedures and standards

In NS&T trace organic analytical procedures, internal standards are added at the start of the procedure and carried through the extraction, cleanup and instrumental analysis. The internal standards when taken through the extraction and clean-up steps and then used for quantification account for analyte losses. Acceptable recovery rates must be higher than 50%. It is the analyst's responsibility to monitor recovery rates and to determine acceptability based on variation of these rates.

#### 4.1.4. Instrument calibration

The results of calibration checks performed at the beginning and end of each typical sample string must be within  $\pm 10\%$  of the accuracy-based value for standards in order to consider the instrument used to be within calibration. The results of spike blank analysis must be within  $\pm 20\%$  of the correct value in order to consider the method to be in a state of control.

#### 4.1.5. Sample quantification

All samples must be quantified within the calibration range. Quantification based on extrapolation is not acceptable.

#### 4.1.6. Method detection limits

Method Detection Limits (MDLs) are calculated and reported annually on a matrix and analyte basis. Since 1989, the method used for calculating MDLs is that used by EPA and is described in detail in the 7/1/88 edition of the Federal Register (Definition and Procedure for the Determination of the Methods Detection Limits - Revision 1.11). If the EPA method is not used or is modified, the procedure used for MDL calculation is described in detail. Separate MDLs are calculated for mussels and oysters. For more information on detection limits see Section 2.3.

#### 4.1.7. Precision

Acceptable limits of precision for organic control materials are  $\pm 30\%$  on average for all analytes, and  $\pm 35\%$  for individual analytes. These limits apply to those materials where the concentrations of the compounds of interest are at least 10 times greater than the MDLs for those compounds. The application of these guidelines in determining the acceptability of the results of the analysis of a sample is a matter of professional judgement on the part of the analyst, especially in cases where the analyte level(s) are near the limit of detection.

Horwitz *et al.* (1980) discussed the inverse relationship between sensitivity and precision and found that, in general, the precision, as a function of concentration, appears to be independent of the nature of the analyte or the analytical technique. The interlaboratory coefficient of variation at the 10 ng/g (ppb) analyte level is expected to be approximately 30% (Figure I.12), and attainment of this level of precision will require the best possible effort on the part of the analyst.

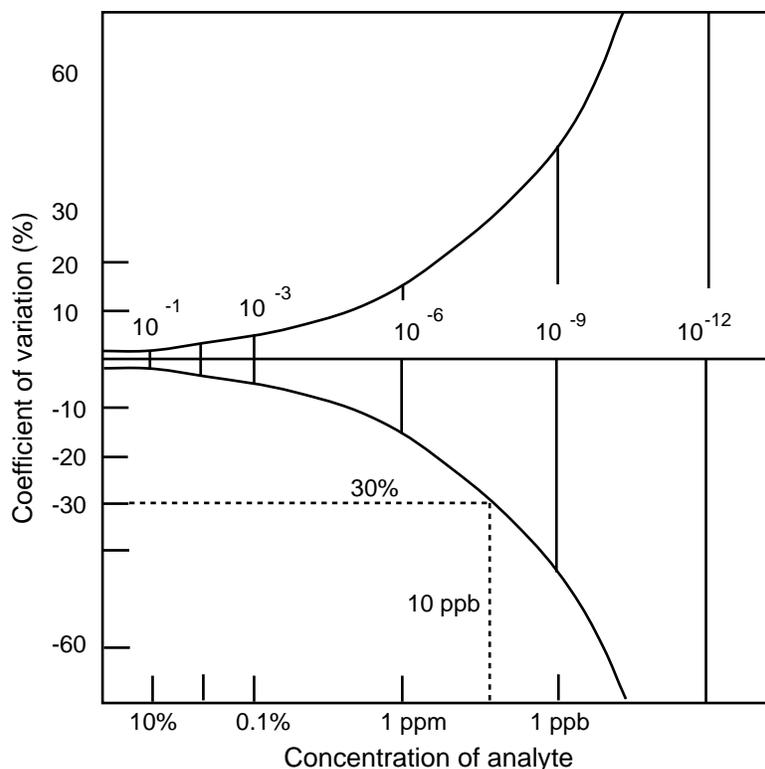


Figure I.12. Interlaboratory coefficient of variation as a function of concentration [Adapted from Horwitz *et al.* (1980)].

#### 4.1.8. Accuracy

Acceptable limits of accuracy are  $\pm 30\%$  of known certified concentrations that are at least 10 times above the limit of detection of an analyte. The certified values and uncertainties found in the NIST Certificate of Analysis for SRMs describe, statistically, the range in which there is a 95% probability the true value is found. The  $\pm 30\%$  range should therefore be calculated as 30% above and below the uncertainties listed in the NIST Certificate of Analysis. For a certified value and uncertainty,  $x \pm y$ , the  $\pm 30\%$  range is

$$(x + y) + [0.3(x + y)]$$

to

$$(x - y) - [0.3(x - y)].$$

#### 4.2. Control samples

A minimum of 8% of an analytical sample string should consist of blanks, reference or control materials, duplicates, and spike matrix samples. The use of control materials does not entirely replace the use of duplicates and spiked matrix samples. A minimum of 2% of the standard inorganic sample string should consist of calibration materials and reference or control materials.

#### 4.3. Data acceptability criteria and archival

The results of the routine analysis of reference and control materials, and blanks; and intercomparison exercises, are reported annually.

#### 4.4. Intercomparison exercises

All the NS&T laboratories are required to participate in the yearly intercomparison exercises which began in 1986. Results of the exercises prior to 1991 are described in Cantillo and Parris (1993) and Valette-Silver (1992), and those of 1991 and 1992 in Cantillo *et al.* (in preparation). The exercise materials are usually sent early in the spring or summer, with complete handling instructions and diskette with data reporting format. The type and matrix of the samples change yearly. If problems are encountered during any of the phases of the intercomparison exercises, the laboratories can contact NRC or NIST for assistance.

The results of the intercomparison exercises are not intended to be a reflection of the absolute capability of a laboratory. Given time and budgetary constraints, the methodology used may not be the one resulting in the lowest detection limits or best precision, rather, it is the one that can be used to generate data of the quality specified by the NS&T Program. Lower detection limits and greater precision are possible by increasing sample size and replication.

Beginning in 1988, the trace organic intercomparison exercises were designed to improve the methodology used by the NS&T laboratories by isolating sources of variability such as sample preparation and extraction. This was done using simple solutions of some of the analytes of interest. Since then, the complexity of the intercomparison exercise materials has increased and the recent exercises also incorporate natural materials.

#### 4.5. Quality Assurance Workshops

The results of the intercomparison exercises are discussed among NIST, NRC, and the participating laboratories during the yearly QA Workshop held in late fall or winter. During such meetings, a consensus is reached between NIST, NRC, NOAA, and the laboratories as to the type of materials that will be used for the following year's intercomparison exercise.

#### 4.6. Development of standard reference and control materials

In response to the needs of the NS&T Program, NOAA has partially funded the production of 8 NIST SRMs and 7 internal standard solutions. The SRMs are based on natural matrices and the calibration solutions are for each of the three chemical classes of analytes at two concentration levels. The latter are used to facilitate the preparation of multipoint calibration curves. The internal standard solutions were prepared at the request of the NS&T contract laboratories and were provided free of charge. These SRMs and control materials have been, and continue to be, used by NS&T contract laboratories to maintain analytical control. The SRMs are available for purchase through NIST.

#### 4.7. NIST trace organic exercises

In response to numerous requests, NIST opened the organic chemical intercomparison exercises in 1993 to laboratories funded by NOAA/NS&T or EPA/EMAP-E. The participants in this effort, titled "NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment", can receive the same materials prepared for the NS&T cooperating laboratories for that year, or can purchase materials from previous exercises. Results received by the

designated deadline are summarized and evaluated by NIST and participants are invited to attend the December NS&T QA Workshop.

#### 4.8. NRC trace element exercises

Certain laboratories participating in the other NOAA/NS&T projects or in EPA/EMAP-E are also participants in the trace element intercomparison exercises. These participants received the same materials. Results received by the designated deadline are summarized and evaluated by NRC. Participants also attend the December NS&T QA Workshop.

### 5. ANALYTICAL PROCEDURES

#### 5.1. Introduction

##### 5.1.1. Trace organics

Analytical protocols for the quantification of the NS&T organic contaminants were developed by MacLeod *et al.* (1984) at the NMFS/NWFSC facilities in Seattle, WA. These methods were prescribed for all NMFS laboratories participating in the NBSP when the NS&T Program began in 1984. Three NMFS laboratories used these methods in 1984: NEFSC, Gloucester, MA; SEFSC, Charleston, SC; and NWFSC, Seattle, WA. The philosophy associated with the development of exacting protocols for the quantification of organic contaminants was that the same analytical methods would increase the likelihood of data being comparable among laboratories. Even though interlaboratory comparisons were initiated at the start of the NS&T Program, it was felt that a method-driven QA and analytical effort for the quantification of organic contaminants was the best way to begin. In 1985, the protocols were updated by MacLeod *et al.*, 1985. This method has been further edited and can be found in Volume I of this document. The NS&T Mussel Watch Project began in 1986. At that time, both the MWP and the NBSP laboratories were allowed to use any analytical method if it could be proven that the proposed alternate procedure was equal to or better than earlier MacLeod *et al.* (1984, 1985) methods.

##### 5.1.2. Major and trace elements

Mandatory protocols were never prescribed for the laboratories quantifying major and trace elements in either the NBSP or MWP. The quantification of elements by the NS&T Program laboratories was perceived to be of a high enough quality that analytical control could be maintained by the use of standard reference materials during the analytical cycle and through interlaboratory comparisons exercises.

#### 5.2. Discussion of analyte limitations

##### 5.2.1. Organics analytes

###### 5.2.1.1. PCBs

###### 5.2.1.1.1. PCB quantitation

In 1984 and 1985, eight PCB congeners (IUPAC numbers 7, 31, 47, 101, 153, 185, 194, and 206) were quantified, each representing a different PCB chlorination level. It was possible to quantify these PCB congeners because the NMFS/NWFSC provided the participating NS&T laboratories with PCB standards containing these congeners. With these 8 congeners

representing PCBs homologues (Cl<sub>2</sub> to Cl<sub>9</sub>), the total concentrations of each PCB homolog were derived. A detailed discussion of the subject can be found in Sericano, 1993. In 1986, the NS&T Program began using a different suite of PCB congeners to quantify concentrations at all 9 PCB chlorination levels. The transition from reporting PCB concentrations by PCB chlorination level to reporting specific congeners began with the quantitation of this set of nine PCB congeners: IUPAC numbers 8, 28, 52, 101, 153, 170, 195, 206, and 209. These congeners, which were supplied by NIST, are some of the major congeners found in commercial Aroclor mixtures and are among those commonly reported in environmental samples (Sericano, 1993). Another consideration was the availability of reasonably pure PCB standards for these congeners. The replacement congeners had slightly different response factors, so concentrations of homologs corresponding to them may also have changed slightly (R. Parris, NIST, Gaithersburg, MD, personal communication, 1993).

In 1987, an additional 9 PCB congeners were added to the list of congeners quantified by the NS&T Program: IUPAC numbers 18, 44, 66, 105, 118, 128, 138, 180, and 187. Beginning in 1988, results from PCB analysis have been reported by all laboratories at the congener (not homolog) level. Planar PCB congeners 77 and 126 were added to the program in 1990, when they were added to the Mussel Watch Project (Table 1.9).

Total PCB concentrations were compared, by the NBSP and MWP laboratories, between the earlier method where PCB congener chlorination levels were used to extrapolate the total PCB concentration of all possible PCB congeners with the "total" PCB concentration found for the sum of the 18 PCB congeners later quantified by the program. Correlations between the total PCB concentration as the sum of chlorination levels and as the sum of 18 congeners showed the sum of chlorination level concentration data to be approximately twice the sum of congener concentrations. Conversion factors are found in Table I.10.

A problem with using one PCB congener to represent all PCB congeners at a given chlorination level is that different congeners have different relative response factors, even within a chlorination level. Thus, a congener that does not have a corresponding standard for use in quantitation might be underestimated or overestimated because of the difference between its relative response factor and that of the corresponding congener (Sericano, 1993; Mullin *et al.*, 1984).<sup>\*</sup> It is for this reason that the NS&T "total" PCBs are now reported as the sum of the quantified congeners and not as the sum of all possible PCB congeners.

#### 5.2.1.1.2. PCB selection

NOAA used a number of criteria to select specific congeners (Table I.9). One criterion was that PCB congeners selected would be ones already being quantified by other scientific organizations, such as laboratories participating with the International Committee of the Exploration of the Seas (ICES) and Community Bureau of Reference (BCR). Three toxic congeners, PCBs 77, 105 and 126, whose structures are similar to that of dioxins or furans, were added at the request of the EPA. Other PCB congeners (PCBs 118, 128, 138, and 170) were added because of their toxicity, their ubiquitousness in the marine environment, or at the suggestion of leaders in the field of PCB quantification.

Clarke *et al.* (1989) divided a sub-set of the possible 209 PCB congeners into four categories of environmental interest. Group 1 includes 3-methylcholanthrene type mixed function oxidase (MFO) inducers along with five mixed-type inducers that have frequently been reported in environmental samples. Of the 8 PCB congeners in this group, the NS&T Program quantifies 6 of them (i.e., PCBs nos. 77, 118, 126, 128, 138, and 170). In the second group are congeners considered to result in phenobarbital type induction. The NS&T PCBs in this category are 101, 153, and 180. Group 3 congeners are weak MFO inducers but occur frequently in environmental

samples. The NS&T PCBs in this category include PCBs 18, 44, 52, and 187. The fourth congener group is found in relatively low concentrations in environmental samples but is of interest because of their potential toxicity. PCB 105, quantified by the NS&T Program, is in this last category.

#### 5.2.1.1.3. PCB coelutions

It should be noted that even though the NS&T currently "quantifies" the concentrations of the planar PCBs 77 and 126, the reported concentrations are not necessarily for individual PCB components. With the analytical methods presently used by NS&T participating laboratories, a number of PCB congeners coelute. PCB 77 is the lesser contributor of the PCB congener pair 77 and 110, and PCB 126 is the lesser contributor of the PCB congener group 126, 129, and 178 (Schulz *et al.*, 1989). Of the twenty PCBs quantified by the NS&T Program, fourteen coelute with other PCB congeners (Table I.11). New clean-up procedures using carbon column chromatography have been developed that allow organic chemists to separate and quantify individual PCB congeners (Sericano *et al.*, 1991).

It should be noted that with the analytical techniques used, it was possible for the more highly chlorinated and later eluting PCBs to have occasionally coeluted with toxaphene. However, GC/ECD response factors (RF) for toxaphene compounds are much lower than for PCB congeners, so PCB concentrations would not be greatly affected unless toxaphene concentrations were high (T. Wade, TAMU/GERG, College Station, TX, personal communication, 1993). Weathered toxaphenes found in environmental samples usually elute later on chromatographic columns than do PCBs (R. Parris, NIST, Gaithersburg, MD, personal communication, 1993).

Swackhamer *et al.* (1987) indicate that in addition to toxaphene other organic contaminants such as heptachlor, dieldrin, DDTs, DDEs, and technical chlordane, may also coelute with PCBs. DDTs and DDEs, however, do not coelute with the PCBs quantified by the NS&T Program (R. Parris, NIST, Gaithersburg, MD, personal communication, 1993).

#### 5.2.1.2. PAHs

Only 18 of the 24 PAHs listed in Table I.2 were quantified at the beginning of the NS&T Program. The remaining six (acenaphthylene, 1,6,7-trimethylnaphthalene, benzo[*b*]- and benzo[*k*]fluoranthene, indeno[1,2,3-*cd*]pyrene, and benzo[*ghi*]perylene) were added in 1988 (NOAA, 1989).

Benzo[*b*]- and benzo[*k*]fluoranthene were quantified as the sum of these two compounds in the 1987 and 1988 Gulf Coast data, and as separate compounds thereafter.

Laboratories using GC/FID (gas chromatography/flame ionization detector) for PAH analysis early in the monitoring program were unable to separate chrysene from triphenylene and these two compounds are usually not separated by DB5 columns (S. Wise, NIST, Gaithersburg, MD, personal communication, 1990, 1993). It was possible to differentiate these analytes when GC/MS (gas chromatography/mass spectrometry) or liquid chromatography was used.

Table I.9. Selection criteria for the 20 polychlorinated biphenyls congeners (S. Wise, NIST, Gaithersburg, MD, personal communication, 1988).

PCB No.	Degree of Chlorination	Major component in environmental mixtures *	Proposed by or justification for
8	2	no	NOAA and suggested by K. Ballschmiter to S. Wise.
18	3	no	ICES <sup>Δ</sup>
28	3	yes	ICES and BCR*. Substituted for PCB 31 in original NOAA calibration solution which coelutes in gas chromatography with PCB 28. PCB 28 is more prominent in environmental samples than PCB 31
44	4	yes	ICES
52	4	yes	ICES and BCR. Substituted for PCB 47 in original NOAA calibration solution. PCB 52 is more prominent than PCB 47 in environmental samples.
66	4	yes	ICES
77	4	no	EPA <sup>†</sup> . Corresponds to 2,3,7,8-tetrachloro- <i>p</i> -di-benzodioxin (TCDD) structure.
101	5	yes	ICES and BCR. In original NOAA calibration solution.
105	5	yes	EPA. Similar to 2,3,7,8-TCDD structure.
118	5	yes	ICES and BCR
126	5	no	EPA. Similar to 2,3,7,8-TCDD structure
128	6	yes	Suggested by K. Ballschmiter to S. Wise
138	6	yes	ICES and BCR
153	6	yes	ICES and BCR. In original NOAA calibration solution.
170	7	yes	ICES
180	7	yes	ICES and BCR. Substituted for PCB 185 in original NOAA calibration solution. PCB 185 degrades in fish.*
187	7	yes	ICES
195	8	no	Substituted for PCB 194 in original NOAA calibration solution because of difficulty in obtaining PCB 194 compound.
206	9	no	ICES
209	10	no	ICES

\*Zell and Ballschmiter, 1980.

<sup>Δ</sup> From list of 34 PCBs recommended for quantitation by International Committee for Exploration of the Seas (ICES).

• From list of seven compounds used by the Community Bureau of Reference (BCR) for quantitation in environmental mixtures.

<sup>†</sup> Added at the request of EPA since the 3,3',4,4'-tetrachlorobiphenyl configuration is important for toxicity considerations.

Table I.10. Summary of statistical analyses of PCB congeners and level of chlorination data.

Level of chlorination (LOC)	Linear Regression Equations		
	Battelle <sup>Δ</sup>	NMFS <sup>◇</sup>	TAMU <sup>◇</sup>
<b>Sediments</b>			
Trichlorobiphenyls	$y = 0.207 + 1.54x$	$y = 3.311 + 1.982x$	
Tetrachlorobiphenyls	$y = -2.09 + 2.45x$	$y = 6.439 + 2.904x$	
Pentachlorobiphenyls	$y = 3.15 + 2.31x$	$y = -0.846 + 3.83x$	
Hexachlorobiphenyls	$y = 0.463 + 1.50x$	$y = 0.345 + 2.191x$	
Heptachlorobiphenyls	$y = -0.434 + 1.73x$	$y = 0.114 + 2.130x$	
Total PCB	$y' = -1.55 + 2.01x'$	$y' = 1.399 + 3.291x'$	$y' = -0.18 + 2.84x'$
<b>Tissues</b>			
	<b>mollusks</b>	<b>fish</b>	<b>oysters</b>
Trichlorobiphenyls	$y = 4.71 + 1.41x$	$y = -0.232 + 1.363x$	
Tetrachlorobiphenyls	$y = 1.80 + 2.51x$	$y = -29.94 + 3.080x$	
Pentachlorobiphenyls	$y = 21.8 + 2.11x$	$y = -14.54 + 2.417x$	
Hexachlorobiphenyls	$y = 9.98 + 1.26x$	$y = -44.22 + 1.837x$	
Heptachlorobiphenyls	$y = 0.92 + 1.73x$	$y = 21.19 + 1.473x$	
Total PCB	$y' = -2.1 + 1.95x'$	$y' = 80.85 + 2.223x'$	$y' = 8.1 + 2.30x'$
<p><math>y</math> = the sum of congener concentrations, determined to be in a particular level of chlorination  <math>y'</math> = the total sum of all levels of chlorination  <math>x</math> = the sum of specific congeners in the sample within the same level of chlorination as represented in the calibration solution  <math>x'</math> = the sum of 18 congeners as identified in the samples</p>			

<sup>Δ</sup>Boehm *et al.* 1988.

<sup>◇</sup>T. O'Connor, NOAA, Silver Spring, MD, personal communication, 1993.

Table I.11. Coeluting PCB congeners.

Target PCB	Target PCB name	Coeluting PCB	Coeluting PCB name
8	2,4'-Dichlorobiphenyl	5 <sup>†</sup>	2,3-Dichlorobiphenyl
18	2,2',5-Trichlorobiphenyl	15 <sup>*</sup>	4,4'-Dichlorobiphenyl
28	2,4,4'-Trichlorobiphenyl	31 <sup>*</sup>	2,4',5-Trichlorobiphenyl
44	2,2',3,5'-Tetrachlorobiphenyl	◇	
52	2,2',5,5'-Tetrachlorobiphenyl	◇	
66	2,3',4,4'-Tetrachlorobiphenyl	95 <sup>*</sup>	2,2',3,5',6-Pentachlorobiphenyl
77 <sup>Δ</sup>	3,3',4,4'-Tetrachlorobiphenyl	110 <sup>*</sup>	2,3,3',4',6-Pentachlorobiphenyl
101	2,2',4,5,5'-Pentachlorobiphenyl	90 <sup>*</sup>	2,2',3,4',5-Pentachlorobiphenyl
105	2,3,3',4,4'-Pentachlorobiphenyl	132 <sup>*</sup>	2,2',3,3',4,6'-Hexachlorobiphenyl
		153 <sup>*</sup>	2,2',4,4',5,5'-Hexachlorobiphenyl
118	2,3',4,4',5-Pentachlorobiphenyl	123 <sup>†</sup>	2',3,4,4',5-Pentachlorobiphenyl
		149 <sup>†</sup>	2,2',3,4',5',6-Hexachlorobiphenyl
126 <sup>Δ</sup>	3,3',4,4',5-Pentachlorobiphenyl	129 <sup>†</sup>	2,2',3,3',4,5-Hexachlorobiphenyl
		178 <sup>†</sup>	2,2',3,3',5,5',6-Heptachlorobiphenyl
128	2,2',3,3',4,4'-Hexachlorobiphenyl	◇	
138	2,2',3,4,4',5'-Hexachlorobiphenyl	163 <sup>*</sup>	2,3,3',4',5,6-Hexachlorobiphenyl
		164 <sup>*</sup>	
153	2,2',4,4',5,5'-Hexachlorobiphenyl	*	see PCB 105
170	2,2',3,3',4,4',5-Heptachlorobiphenyl	190 <sup>*</sup>	2,3,3',4,4',5,6-Heptachlorobiphenyl
180	2,2',3,4,4',5,5'-Heptachlorobiphenyl	◇	
187	2,2',3,4',5,5',6-Heptachlorobiphenyl	182 <sup>*</sup>	2,2',3,4,4',5,6'-Heptachlorobiphenyl
		159 <sup>*</sup>	2,3,3',4,5,5'-Hexachlorobiphenyl
195	2,2',3,3',4,4',5,6-Octachlorobiphenyl	208 <sup>†</sup>	2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl
206	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	◇	
209	Decachlorobiphenyl	◇	

<sup>†</sup> Results from Schulz *et al.*, 1989, using a 50 m SE-54 fused silica column.

\* Results from Schantz *et al.*, 1993, using a 60 m DB5 column. This column type was typically used to quantify PCBs and chlorinated pesticides in the NS&T Program. PCB 153 closely elutes with PCB105 and PCB 132 while PCBs 105 and PCB 132 almost coelute.

◇ Single gas chromatographic peak, no coeluting congeners significantly present in Aroclors or Clophen mixtures (Schulz *et al.*, 1989).

Δ Planar PCBs.

## 5.2.2. Inorganic analytes

### 5.2.2.1. Thallium

Thallium was quantified through 1989 of the NS&T Program. Thallium was difficult to quantify because of its low environmental concentrations and so its analysis was discontinued.

### 5.2.2.2. Antimony

As with thallium, antimony was difficult to quantify using existing technologies and for this reason less emphasis was placed on its quantitation after 1990.

Table I.12. Alkylated PAHs quantified in the NS&T Mussel Watch Project in 1993.\*

---

C1 - Naphthalenes	Dibenzothiophene
C2 - Naphthalenes	C1 - Dibenzothiophenes
C3 - Naphthalenes	C2 - Dibenzothiophenes
C4 - Naphthalenes	C3 - Dibenzothiophenes
C1 - Fluorenes	C1 - Fluoranthene + pyrenes
C2 - Fluorenes	C1 - Chrysenes
C3 - Fluorenes	C2 - Chrysenes
C1 - Phenanthrenes + anthracene	C3 - Chrysenes
C2 - Phenanthrenes + anthracene	C4 - Chrysenes
C3 - Phenanthrenes + anthracene	
C4 - Phenanthrenes + anthracene	

---

\* These are also quantified by the NWFSC.

#### 5.2.2.3. Selenium

Quantification of Se has been performed since the inception of the NS&T Program. Sediment Se data for the majority of the MWP sites was obtained only during the first two years of the Project (1986 and 1987). Selenium is ubiquitous in marine sediments yet because of the high detection limits of the laboratory analyzing samples from California and Hawaii, this contaminant was frequently reported to be below the detection limit during these two years.

#### 5.2.2.4. Tin

Similar considerations for Se also apply for Sn. Beginning in 1988, analysis of mollusks for Se for California and Hawaii samples was performed by Battelle and Battelle began performing Sn analyses for all West Coast sites in 1989, see Table I.8.

#### 5.3. Analyte additions

Tributyltin is a powerful biocide used in marine paints. In 1987, tributyltin and its metabolites (mono-, di-, and a by-product, tetrabutyltin) were quantified in selected sediments and mollusks from MWP sites. Mollusk butyltin quantification became a regular part of the MWP in 1988. Butyltin quantification became a regular part of the NBSP in 1988 (Krone *et al.*, 1991).

Since it is not possible to quantify all contaminants in all samples, broad scan analyses were performed on 10% of all the MWP samples for otherwise unquantified polar aromatic and halogenated hydrocarbons. To ensure that the possibility exists for retrospective analyses for over-looked contaminants, the NS&T Program maintains a Specimen Bank where sediment, and fish and mollusk tissues samples are stored in liquid nitrogen freezers (Lauenstein, *et al.*, 1987).

In 1989, eight contemporary pesticides were analyzed at three MWP East Coast sites. These were toxaphene, endosulfan I and II, atrazine, propanil, methyl parathion, carbaryl and alachlor. Of these only endosulfan I was above the detection limit at two sites. In 1992, alkylated PAHs (Table 1.12) were quantified as part of the MWP along the Gulf Coast with all MWP sites quantified for alkylated PAHs in 1993. In 1993, contemporary pesticides to be quantified at the Gulf Coast MWP sites include endosulfan I and II, endosulfan sulfate, pentachlorophenol, chlorpyrifos, and toxaphene.

Table I.13. National Benthic Surveillance Project matrices and parameters measured.

	Benthic Fish			Sediments
	Stomachs	Liver	Bile	
Trace and Major Elements	●	●		●
PAHs	●			●
PCBs	●	●		●
Chlorinated Pesticides	●	●		●
PAH Metabolites			●	
Coprostanol				●
<i>Clostridium perfringens</i>				●

#### 5.4. National Benthic Surveillance Project analytical methods

The NBSP was initiated in 1984 with the annual collection and analyses of benthic fish and associated sediments from 50 coastal sites in the coastal U.S. including, Alaska. Analyses were performed for the analytes found in Tables I.1 - I.5. Because fish can metabolize PAHs in their livers, fish bile was analyzed for PAH metabolites. Sample matrices and the contaminants that were quantified from them are presented below in Table I.13.

Since the inception of the NBSP, all West Coast collections and analyses have been performed by the NMFS/NWFSC. During the years 1984 through 1986, NEFSC, Sandy Hook, NJ, performed all trace element analyses on samples from Maine through Virginia, while the SEFSC in Beaufort, NC has performed all trace element analyses for the Southeast (North Carolina through Texas). For the years 1984 through 1986 the NEFSC in Gloucester, MA was responsible for organic analyses on samples from the Northeast. The SEFSC in Charleston, SC was responsible for organic analyses on samples from the Southeast, for the years 1984 through 1987. Since 1988, all organic analyses nationwide have been performed by the NWFSC. In addition, since 1987, NWFSC has performed all trace element analyses for samples from the Northeast and West Coasts. Elemental analyses of the Southeast and Gulf Coast samples continue to be performed by SEFSC. Analytical methods used are listed in Table I.14.

##### 5.4.1. Inorganic analyses

###### 5.4.1.1 Sediments

Laboratories responsible for inorganic analyses, and laboratory changes over time are shown in Table I.7. Table I.15 lists analytical methods used by the NMFS laboratories to quantify major and minor trace elements in sediments and fish tissues.

###### 5.4.1.1.1. NEFSC sediment elemental analyses

The method used by NEFSC for the analyses of major and trace elements is described in detail in Zdanowicz and Finneran (Volume III, this document). Briefly, 450 mg of dried, homogenized sediment underwent complete dissolution using concentrated nitric acid, hydrochloric acid and hydrofluoric acid at high temperature in a Teflon bomb in a conventional oven. QA samples

including reagent blanks, control materials and reference materials were included as part of each analytical sample string. Calibration curves using standards of four different concentrations, including zero, were used, and at least three replicate determinations for each concentration were used to calculate the calibration curve using linear, least-squares regression. The correlation coefficients were typically >0.98 for a well-behaved analysis. Element quantitation methods are listed in Table I.15.

#### 5.4.1.1.2. SEFSC sediment elemental analyses

The method used by the SEFSC for the analyses of major and trace elements is described in detail in Evans and Hanson (Volume III, this document). Approximately 120 mg of dried, homogenized sediment underwent complete dissolution using a mixture of hydrofluoric, hydrochloric, and nitric acids. Early in the project, sample sediments were placed in a Teflon bomb and digested in an oven at high temperatures. Later, samples were placed in Teflon vials and microwave ovens were used as the heat source for the digestion process. QA samples including reagent blanks, control materials and reference materials were included as part of each analytical string. Calibration curves using standards of four different concentrations including zero were used, and at least two replicate determinations for each concentration were used to calculate the calibration curve using least squares regression. Element quantitation methods are listed in Table I.15.

#### 5.4.1.1.3. NWFSC sediment elemental analyses

In 1984, NWFSC began analyzing surficial sediments collected on the West Coast as part of the NBSP. Since 1987, sediments from the Northeast Atlantic Coast, Maine to and including Virginia, have also been quantified by NWFSC. Detailed descriptions of the methods are available in Robisch and Clark (Volume III, this document). Briefly, approximately 250 mg of dried homogenized sediment underwent complete dissolution using concentrated aqua regia and hydrofluoric acid. Digestion occurred in a Teflon bomb by irradiation in a microwave oven. Calibration curves using standards of four different concentrations, including zero, were calculated using the non-linear equations from the atomic absorption spectrophotometer on-line computer. Instrumental quantitation techniques are listed in Table I.15.

### 5.4.1.2 Tissue

#### 5.4.1.2.1. NEFSC tissue elemental analyses

The NEFSC laboratory quantified trace elements in fish liver samples for the Northeast U.S. Coast for the NBSP years 1984 through 1986. Complete descriptions of these methods are found in Zdanowicz and Finneran (Volume III, this document). One gram of tissue was taken from one fish or a composite 1 g sample was prepared using livers of 10 fish. No more than 3 g wet weight of a dried composite liver homogenate underwent complete dissolution using concentrated nitric acid and subsequent heating in a Teflon bomb. QA samples, including three reagent blanks and three standard reference material samples, were included as part of each analytical string. Standards of four different concentrations, including zero, were used, and at least three replicate determinations were made for each concentration. A standard was analyzed at the start of each analytical string and after every three samples. The calibration curve was calculated using linear regression of the standards data. Instrumental quantitation techniques are listed in Table I.15.

Table I.14. Analytical methods used in the National Benthic Surveillance Project.

	Matrix	Units (dry wt.)	Method	Reference <sup>◇</sup>
<b>Organic compounds</b>				
Pesticides, PCBs	Tissue	ng/g	GC/ECD	MacLeod <i>et al.</i> , 1985 Sloan <i>et al.</i>
	Sediment	ng/g	GC/ECD	MacLeod <i>et al.</i> , 1985 Sloan <i>et al.</i>
PAHs	Stomach contents	ng/g	GC/FID/MS	MacLeod <i>et al.</i> , 1985 Varanasi <i>et al.</i> , 1989 Sloan <i>et al.</i>
	Sediment	ng/g	GC/FID/MS	MacLeod <i>et al.</i> , 1985 Varanasi <i>et al.</i> , 1989 Sloan <i>et al.</i>
PAH Metabolites	Fish bile (wet weight)	ng equivalents/g	HPLC/FID	Krahn <i>et al.</i> , 1986 Varanasi <i>et al.</i> , 1989
Coprostanol	Sediment	ng/g	GC/FID	MacLeod <i>et al.</i> , 1985 Sloan <i>et al.</i>
<b>Major and trace elements</b>				
Al <sup>†</sup> , Ag, Al, As, Cd, Cr, Ni, Pb, Sb, Se, Sn <sup>*</sup> , Tl	Tissue	μg/g	GFAA	Evans and Hanson Robisch and Clark Zdanowicz and Finneran
Fe, Mn, Cu <sup>Δ</sup> , Zn	Tissue	μg/g	FAA	same authors
Hg	Tissue	μg/g	CVAA	same authors
Si, Al, Fe	Sediment	%	FAA	same authors
Cr <sup>•</sup> , Zn, Mn	Sediment	μg/g	FAA	same authors
Ag, As, Cd, Cu <sup>#</sup> , Ni, Pb, Sb, Se <sup>▼</sup> , Sn <sup>*</sup> , Tl	Sediment	μg/g	GFAA	same authors
Hg	Sediment	μg/g	CVAA	same authors
<b>Other parameters</b>				
<i>C. perfringens</i>	Sediment	spores/g	Plate count	Bisson and Cabelli, 1979

<sup>◇</sup>All organic methods with the exception of Krahn *et al.* (1986), and Varanasi *et al.* (1989) can be found in Volume IV, and all trace element methods can be found in Volume III, this document.

<sup>†</sup> Not quantified by SEFSC.

<sup>\*</sup> Robisch and Clark also used HAA.

<sup>Δ</sup> Robisch and Clark used GFAA.

<sup>•</sup> Evans and Hanson used GFAA.

<sup>#</sup> Zdanowicz and Finneran used FAA.

<sup>▼</sup> Evans and Hanson used HAA.

Table I.15. National Benthic Surveillance inorganic instrumental analysis methods.

	NEFSC		NWFSC		SEFSC	
	Sediments 1984 - 1986	Tissues	Sediments 1984 - 1992	Tissues	Sediments 1984 - 1992	Tissues
Al	FAA	GFAA	FAA	GFAA	FAA	----
Si	FAA	----	FAA	----	FAA	----
Cr	FAA	GFAA	FAA	GFAA	GFAA	GFAA
Mn	FAA	FAA	FAA	FAA	FAA	FAA
Fe	FAA	FAA	FAA	FAA	FAA	FAA
Ni	GFAA	GFAA	GFAA	GFAA	GFAA	GFAA
Cu	FAA	FAA	GFAA	GFAA	GFAA	FAA
Zn	FAA	FAA	FAA	FAA	FAA	FAA
As	GFAA	GFAA	GFAA	GFAA	GFAA	GFAA
Se	GFAA	GFAA	GFAA	GFAA	HAA	GFAA
Ag	GFAA	GFAA	GFAA	GFAA	GFAA	GFAA
Cd	GFAA	GFAA	GFAA	GFAA	GFAA	GFAA
Sn	GFAA	GFAA	GFAA/HAA	GFAA/HAA	GFAA	GFAA
Sb	GFAA	GFAA	GFAA	GFAA	GFAA	GFAA
Hg	CVAA	CVAA	CVAA	CVAA	CVAA	CVAA
Tl	GFAA	GFAA	GFAA	----	GFAA	GFAA
Pb	GFAA	GFAA	GFAA	GFAA	GFAA	GFAA

FAA - Flame Atomic Absorption; GFAA - Graphite Furnace Atomic Absorption; CVAA - Cold Vapor Atomic Absorption; HAA - Hydride Generation Atomic Absorption.

#### 5.4.1.2.2. SEFSC tissue elemental analyses

The SEFSC laboratory has quantified trace elements in fish liver samples for southeast and Gulf Coast samples since the inception of the NBSP. Complete descriptions of the methods summarized below are in Evans and Hanson (Volume III, this document). In the years 1984 to 1987, analyses were made on 1 g samples of fish livers of individual fish. Since 1988, composite samples were prepared using 1 g aliquots of the liver of 10 fish. Approximately 1 g wet weight of a composite liver homogenate underwent complete dissolution using concentrated nitric acid and subsequent heating in a Teflon bomb by irradiation in a microwave oven. QA samples, including reagent blanks, control materials and reference materials, were included as part of each analytical string. Calibration curves using standards of four different concentrations including zero were used, and at least two replicate determinations for each concentration were used to calculate the calibration curve. Instrumental quantitation techniques are listed in Table I.15.

#### 5.4.1.2.3. NWFSC tissue elemental analyses

NWFSC began analyzing fish liver tissues of specimens collected on the West Coast on an annual basis in 1984, and has performed elemental analyses for the northeast samples since 1987. The NWFSC analytical methods are similar to those used by SEFSC in that fish tissues were completely dissolved and the complete dissolution was accomplished using Parr bombs. The NWFSC method is described in detail in Robisch and Clark (Volume III, this document).

Briefly, approximately 1 g wet weight of an individual or composite liver homogenate underwent complete dissolution using concentrated nitric acid and subsequent heating in a Teflon bomb by irradiation in a microwave oven. Hydrogen peroxide was added to complete the oxidation process. Composite samples were prepared using 1 g each from the livers of 10 fish. QA samples including reagent blanks, control materials and SRMs were included as part of each analytical sample string. Instrumental quantitation techniques are listed in Table I.15.

Calibration curves using standards of five different concentrations, not including zero, were used. Three replicate injections were analyzed for each standard. Calibration curves were routinely calculated using non-linear equations from the on-line computer.

#### 5.4.2. Organic analyses

##### 5.4.2.1. General methods for sediments and tissues

The NMFS/NWFSC National Analytical Facility developed techniques for the extraction, separation, and quantification of PAHs, PCBs, and chlorinated pesticides in both marine tissues and sediments (MacLeod *et al.* 1984, MacLeod *et al.* 1985). These techniques were the starting point for organic analyte quantification for both the National Benthic Surveillance and Mussel Watch Projects. The 1985 MacLeod *et al.* document was edited and expanded and is included in Volume IV, of this document, and is summarized below.

The sample was thawed (if frozen) and 10 g of homogenized sediment or 3 g of homogenized tissue composite were weighed. Samples were combined with anhydrous sodium sulfate and extracted 3 times with dichloromethane. The dichloromethane solvent in the sample extracts was then replaced by hexane. Silica/alumina column chromatography was used to separate the extracted analytes into three fractions: the saturated hydrocarbons and possibly hexachlorobenzene (SA1), aromatic hydrocarbons and chlorinated pesticides (SA2), and coprostanol (SA3) (Figure I.13). The aliphatic (saturated) hydrocarbons were not quantified. The second eluted fraction (SA2) was further separated using a Sephadex column. Two sample fractions were eluted: the lipids and biogenic material, and the aromatic and chlorinated pesticides. Only the second fraction was used for quantitation. Chlorinated compounds were quantified using GC/ECD, and the aromatic compounds were quantified using GC/FID. Coprostanol was isolated and quantified as a tracer of human/mammalian fecal matter in sediments and was quantified with the use of GC/FID or GC/MSD. After 1990, the bacterium *Clostridium perfringens* was quantified by both monitoring projects in sediments to determine the level of fecal coliform resulting from mammalian waste.

Method modifications incorporating the use of high performance liquid chromatography (HPLC) to improve the extraction of tissues and sediments were implemented in 1987, when NWFSC became responsible for the quantification of organic contaminants in all samples of the NBSP (Krahn *et al.*, 1988). Briefly, the sediment or tissue extracts were obtained as described in MacLeod *et al.* (Volume IV, this document), then tissue extracts were filtered through silica gel/alumina and sediment extracts through glass wool (Figure I.14). An aliquot of the extract was chromatographed on a 100-Å size-exclusion HPLC column using dichloromethane as the mobile phase. The fraction containing aromatic hydrocarbons and chlorinated hydrocarbons was collected. The concentrated extract was analyzed using GC/ECD to quantify chlorinated hydrocarbons, and GC/FID or GC/MSD to quantify aromatic hydrocarbons.

HPLC methods for separation of sediment extracts for coprostanol determination were also developed by Krahn *et al.* (1989). Coprostanol was quantified as the trimethylsilyl ether using GC/FID.

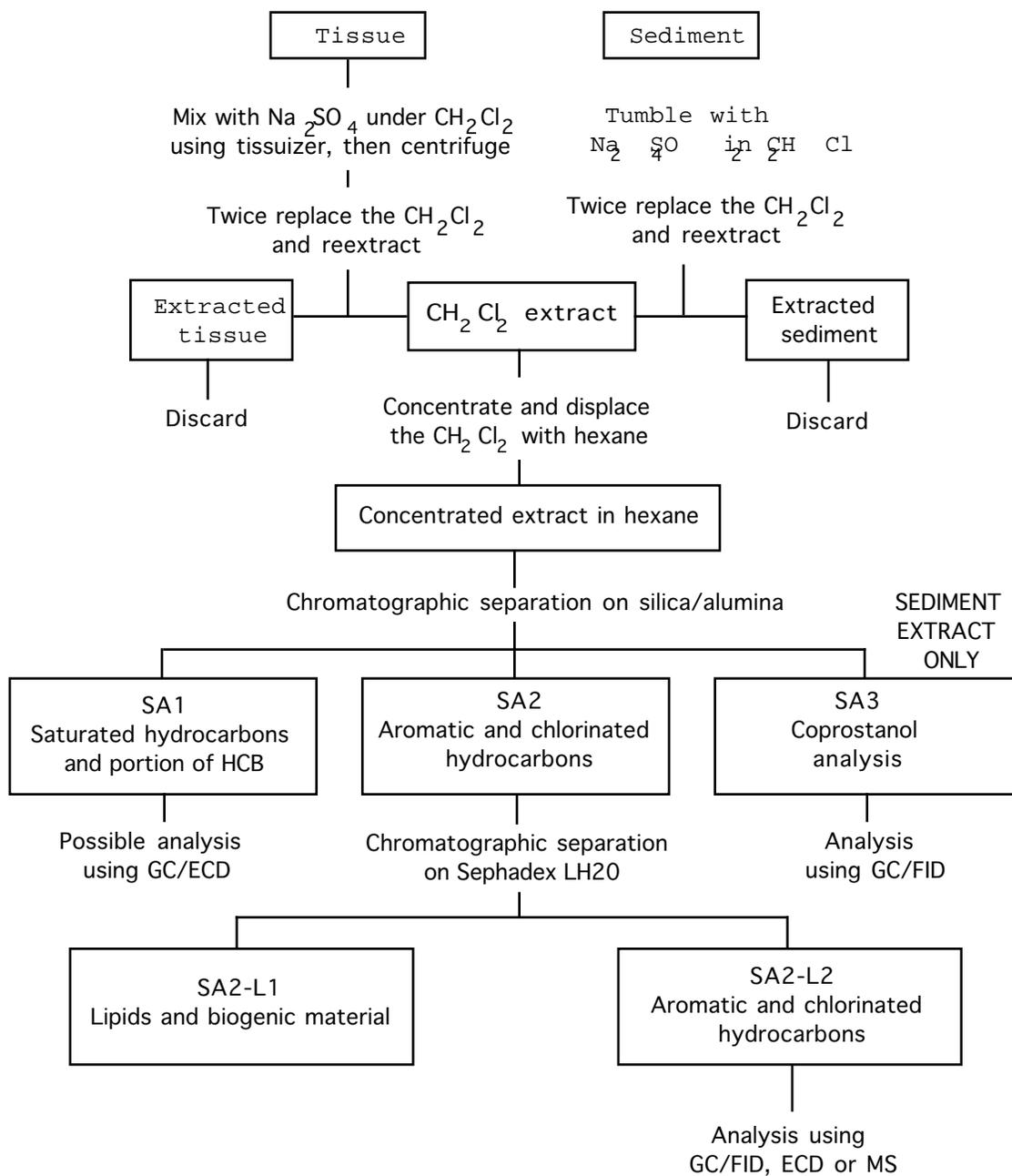


Figure I.13. Scheme of analyses of sediment and tissues (MacLeod *et al.*, 1985).

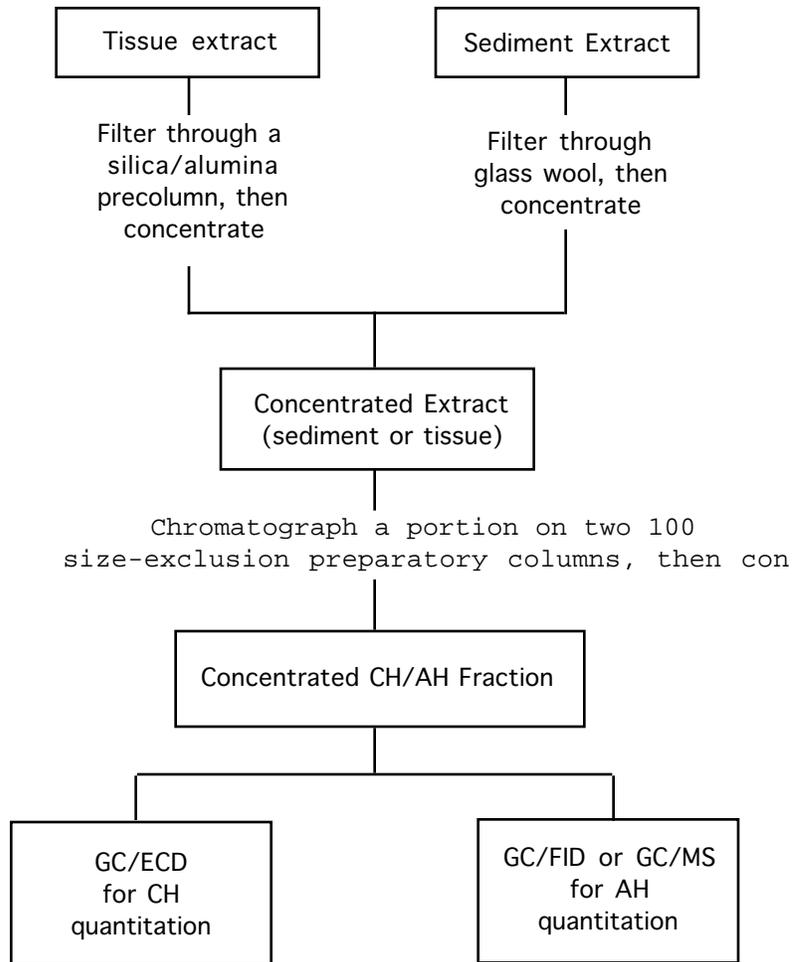


Figure I.14. Flow diagram of extract cleanup procedure (from Krahn *et al.*, 1988).

#### 5.4.2.2. Specific Matrices

##### 5.4.2.2.1. Bile

PAHs were not quantified in fish liver samples because fish are able to metabolize PAHs and excrete the metabolites into the bile duct. However, quantification of PAH metabolites in fish bile is a way to determine the degree to which fish have been exposed to PAHs. Quantitation of bile PAH metabolites began in 1985 and the method was documented in Krahn *et al.* (1986). Briefly, bile was collected from enough fish to obtain a single pooled sample of 3 mL. A 5- $\mu$ L aliquot of the bile composite sample was injected into an HPLC column with a fluorescence detector set at benzo[*a*]pyrene and naphthalene absorption wavelengths. Calibration solutions and duplicate samples were used as part of the sample string.

Table I.16. Mussel Watch Project inorganic instrumental analysis methods used by Battelle.

	1986-1990		1991-1992	
	Tissues	Sediments	Tissues	Sediments
Al	GFAA	XRF	GFAA/ICP-MS	ICP-MS/XRF
Si	XRF	XRF	XRF	XRF
Cr	GFAA	XRF	GFAA/ICP-MS	ICP-MS/XRF
Mn	XRF	XRF	XRF	XRF
Fe	XRF	XRF	XRF	XRF
Ni	GFAA	XRF	GFAA/ICP-MS	XRF
Cu	XRF	XRF	XRF	XRF
Zn	XRF	XRF	XRF	XRF
As	XRF	XRF	XRF	XRF
Se	XRF	GFAA/XRF	XRF	XRF/GFAA
Ag	GFAA	GFAA	GFAA/ICP-MS	GFAA/ICP-MS
Cd	GFAA	GFAA/XRF	GFAA/ICP-MS	GFAA/XRF
Sn	HAA	HAA	HAA/ICP-MS	HAA/ICP-MS
Sb	GFAA/NAA <sup>Δ</sup>	GFAA/NAA <sup>Δ</sup>	GFAA/ICP-MS	GFAA/ICP-MS
Hg	CVAA	CVAA	CVAA	CVAA
Tl	GFAA	GFAA	GFAA	GFAA
Pb	GFAA	XRF	GFAA/ICP-MS	XRF

FAA - Flame Atomic Absorption; GFAA - Graphite Furnace Atomic Absorption; CVAA - Cold Vapor Atomic Absorption; HAA - Hydride Generation Atomic Absorption; <sup>Δ</sup> NAA (Neutron Activation analysis) used in 1986 and 1987.

#### 5.4.2.2.2. Liver

Liver tissue was the most frequently quantified matrix other than sediments in the NBSP. For the organic analyses of liver samples of fish collected along the Pacific and northeast Atlantic Coasts, three composite samples of ten livers each are used. Since target fish collected on the southeast Atlantic and Gulf Coasts are smaller than those collected on the West Coast, composites were prepared from between 10 to 20 livers.

#### 5.4.2.2.3. Muscle

The analytical method for the quantification of fish muscle is similar to that used for the quantification of fish liver tissue.

#### 5.4.2.2.4. Stomach contents

Chlorinated organic compounds, aromatic hydrocarbons, and trace element analyses were performed on fish stomach contents from selected sites. One measurement per site results from the analysis of a single composite of the contents of ten individual stomachs.

#### 5.4.2.2.5. Sediments

There is a difference between methods used by laboratories to quantify sediment organic contaminants. The NBSP quantified organic contaminants in sediments after the overlying water was first decanted and the residual water was removed using a centrifuge. The MWP for the East and West Coasts extracted organic contaminants after first decanting the overlying water, while the Gulf Coast MWP laboratory freeze dried sediments without first removing the water.

#### 5.5. Mussel Watch Project

Three organizations have been primarily involved in sample collection and analysis: TAMU GERG for the Gulf Coast, and Battelle Ocean Sciences for the East and West Coasts. Science Applications International Corp. was active in the Project from 1986 through 1989 as a subcontractor to Battelle. Laboratory responsibilities have changed over the years and this can be seen in Table I.8.

Each laboratory chose its own method for quantitation of major and trace elements. When the Program began, three laboratories were performing major and trace element analyses: TAMU; Battelle, Sequim, WA; and SAIC, La Jolla, CA. The SAIC laboratory performed collection and analyses of samples for Battelle in California and Hawaii.

The suggested method for quantitation of trace organic contaminants was that of MacLeod *et al.* (Volume IV, this document). Since the NS&T QA Project was already using a performance based approach, the Mussel Watch laboratories were not constrained to those methods, though Battelle and SAIC did begin sample analyses using the methods prepared by MacLeod *et al.*, 1984.

#### 5.5.1. Inorganic analyses

##### 5.5.1.1. Battelle sediment and mollusk elemental analyses

##### 5.5.1.1.1. Sediment analyses

These methods are described in detail by Crecelius *et al.* (Volume III, this document). Briefly, approximately 500 mg of homogenized dried sediment underwent complete dissolution using nitric acid and perchloric acid digestion at high temperature in a Teflon bomb using a conventional oven. Hydrofluoric acid and further heating was used to assure complete dissolution of silica. In 1991, Battelle began using ICP-MS to quantitate elements. Before analysis by ICP-MS, an aliquot of digestate (as above) was dried at high temperature to remove the chloride and fluoride, redissolved in nitric acid, and again heated to dryness. The dried digestate was then dissolved with nitric acid and water. Sample dissolution is not required for the use of X-ray fluorescence. Samples were ground, when necessary, and homogenized prior to analysis. Instrumental quantitation techniques are listed in Table I.16.

Table I.17. Mussel Watch Project inorganic instrumental analysis methods used by SAIC.

Element	Tissues			Sediments		
	1986	1987	1988-1989	1986	1987	1988-1989
Al	GFAA/FAA	GFAA/FAA	GFAA	FAA	FAA	GFAA
Si	COLOR	COLOR	----	COLOR	COLOR	----
Cr	GFAA	GFAA	GFAA	GFAA	GFAA	GFAA
Mn	GFAA/FAA	GFAA/FAA	----	FAA	GFAA/FAA	----
Fe	FAA	GFAA/FAA	FAA	FAA	GFAA/FAA	FAA
Ni	GFAA	GFAA	GFAA	GFAA	GFAA	GFAA
Cu	GFAA/FAA	GFAA/FAA	GFAA	GFAA	GFAA	GFAA
Zn	FAA	FAA	FAA	FAA	FAA	FAA
As	GFAA	GFAA	GFAA	GFAA	GFAA	GFAA
Se	GFAA	GFAA	GFAA <sup>Δ</sup>	GFAA	GFAA	GFAA <sup>Δ</sup>
Ag	GFAA	GFAA	GFAA	GFAA	GFAA	GFAA
Cd	GFAA/FAA	GFAA	GFAA	GFAA	GFAA	GFAA
Sn	GFAA	GFAA	◇	GFAA	GFAA	◇
Sb	GFAA	GFAA	----	GFAA	GFAA	----
Hg	CVAA	CVAA	CVAA	CVAA	CVAA	CVAA
Tl	GFAA	GFAA	----	GFAA	GFAA	----
Pb	GFAA	GFAA	GFAA	GFAA	GFAA	GFAA

FAA - Flame Atomic Absorption; GFAA - Graphite Furnace Atomic Absorption; COLOR - Colorimetry. <sup>Δ</sup> All Year 4 samples analyzed for Se and Sn by Battelle. <sup>◇</sup> All Year 3 samples analyzed for Sn by Battelle.

#### 5.5.1.1.2. Tissue analyses

These methods are described in detail by Crecelius *et al.* (Volume III, this document). Briefly, approximately 500 mg of freeze dried and homogenized oyster or mussel tissue underwent complete dissolution using nitric acid and perchloric acid in a Teflon digestion bomb using a conventional oven. Microwave digestion of tissue samples was also used to reduce the time required for digestion. In this procedure, approximately 300 mg of freeze dried and homogenized oyster or mussel tissue underwent dissolution by mixing with nitric acid in a Teflon digestion bomb and irradiating the mixture in a microwave oven. QA samples, including reagent blanks, control materials and SRMs, were included as part of each analytical sample string. Calibration curves using standards of four different concentrations, including zero, were used. At least three replicate determinations were made for each standard concentration. The calibration curve was calculated using least-squares regression. Instrumental quantitation techniques are listed in Table I.16.

#### 5.5.1.2. SAIC sediment and mollusk elemental analyses

The analysis of sediment samples collected in California and Hawaii from 1986 to 1989 was performed by SAIC, with exceptions noted in Table 17. The following information is taken from Peven *et al.*, (Volume III, this document), Boehm *et al.* (1987), Boehm *et al.* (1988), and Freitas *et al.* (1989).

Table I.18. Mussel Watch Project inorganic instrumental analysis methods used by TAMU.

Element	Tissue	Sediment	Element	Tissue	Sediment
Al	FAA	NAA	Se	GFAA	GFAA
Si			Ag	GFAA	GFAA
Cr	GFAA	NAA	Cd	GFAA	GFAA
Mn	FAA	NAA	Sn	GFAA	GFAA
Fe	FAA	NAA	Sb		
Ni	GFAA	GFAA	Hg	CVAA	CVAA
Cu	FAA/GFAA	FAA/GFAA	Tl	----	----
Zn	FAA	FAA	Pb	GFAA	GFAA
As	GFAA	GFAA			

FAA - Flame atomic absorption spectrophotometry; NAA - Neutron activation analysis.

#### 5.5.1.2.1. Sediment analysis

For the quantification of all major and trace elements except Hg, approximately 0.2 g of dried homogenized sediment underwent complete dissolution using nitric acid and hydrochloric acid, with subsequent heating in a 95°C water bath for 2 hr. Hydrofluoric acid was added and then samples were heated in an autoclave for 2 hr. For Hg analysis, approximately 1 g of wet sediment was dissolved using nitric acid and sulfuric acid and heating at low temperature. Analyte quantitation techniques are those presented in Table I.17.

#### 5.5.1.2.2. Tissue analysis

For the quantification of all major and trace elements except Hg, approximately 1 g of dried homogenized mollusk tissue underwent complete dissolution using nitric acid. Samples were first allowed to stand at room temperature, then heated in a water bath, and finally heated in an autoclave. For Hg analysis, approximately 2 g of wet homogenized mollusk tissue was dissolved using nitric acid and sulfuric acid and heating at low temperature. Potassium permanganate was added to complete the process. Analyte quantitation techniques are those presented in Table I.17.

#### 5.5.1.3. TAMU sediment and mollusk elemental analyses

Complete descriptions of the methods used by TAMU GERG are found in Taylor and Presley (Volume III, this document) and Brooks *et al.* (1990).

##### 5.5.1.3.1. Sediment analysis

Briefly, approximately 0.2 g of dried homogenized sediment underwent complete dissolution using nitric acid and perchloric acid in a Teflon bomb and heating in a conventional oven. Hydrofluoric acid, boric acid and further heating were used to assure complete dissolution of silicates. Analyte quantitation techniques are those presented in Table I.18.

##### 5.5.1.3.2. Tissue analysis

Briefly, approximately 0.2 g of dried homogenized mollusk tissue underwent complete dissolution using nitric acid and perchloric acid and heating using a conventional oven.

Calibration curves using standards of 4 different concentrations, including zero, were used. A least squares regression was used to derive the calibration curve. Analyte quantitation techniques are those presented in Table I.18.

#### 5.5.2. Organic analyses

From 1986 to 1989 inclusive, sediment sample analyses performed by Battelle and SAIC were accomplished using modifications of the method of MacLeod *et al.* (Volume IV, this document), while TAMU used their existing laboratory's analytical methods.

##### 5.5.2.1 Battelle and SAIC sediment and mollusk organic analyses

The analytical methods used by Battelle and SAIC are described in detail in Peven and Uhler (Volume IV, this document), as well as in earlier documents: Battelle (1986), Battelle (1987b), Battelle (1988), and Hillman *et al.*, 1992.

For the extraction of tissues for trace organic analysis, 50 g of sodium sulfate was added to approximately 15 g of the bulk homogenized tissue. The tissue was then extracted three times with dichloromethane on a Tisumizer. Sample was removed from the extracted tissues with a centrifuge.

Fifty grams of sediment wet weight were dried with anhydrous sodium sulfate and extracted with dichloromethane and acetone on a shaker table. The extracted sample was removed with a centrifuge. Samples at SAIC were extracted with methanol and dichloromethane with the use of tumbler and centrifuge.

Since the beginning of the project, Battelle and SAIC used GC/ECD for the quantification of chlorinated contaminants.

During the first year of the MWP, Battelle quantified PAHs using GC/FID. In 1987, Battelle began to quantify PAHs using GC/MS in the full scan mode. While the detection limits for MS are not as low as those of FID, MS allows for contaminant identification as well as quantification. In 1988, the use of MS with selected ion monitoring (SIM) was instituted. This step was taken to improve sensitivity of the GC/MS method, since many of the PAH compounds found in bivalve tissues were very near the GC/MS full-scan detection limit (Peven and Uhler, Volume IV, this document).

For the first 3 years that SAIC participated in the NS&T MWP, GC/FID was used for the quantification of PAHs. In 1989, SAIC also converted to the use of GC/MS in the selected ion mode.

Three-point calibration curves were calculated prior to analysis of each analytical sample string.

##### 5.5.2.3. TAMU sediment and mollusk organic analyses

The TAMU GERG analytical methods are described in detail by Wade *et al.* (Volume IV, this document). Briefly, approximately 10 g of homogenized wet weight tissue were extracted using anhydrous sodium sulfate and dichloromethane. The extract was concentrated and purified using a silica/alumina column to remove matrix interferences. During the years 1986, 1987, and 1988 further purification was performed using Sephadex. High performance liquid chromatography (HPLC) has been used since 1989, to reduce matrix interferences. Concentrated extract was analyzed by GC/MS in the selected ion mode for PAHs and by GC/ECD for chlorinated hydrocarbons.

Two methods were employed to extract sediment samples (Wade *et al.*, Volume IV, this document). For the years 1986 through 1988, 10 g of dry sediment were extracted with dichloromethane using a roller table. The method was a modification from the techniques of MacLeod *et al.* During the years 1989 through 1992, 10 g (dry weight) of freeze-dried sediment was Soxhlet-extracted with CH<sub>2</sub>Cl<sub>2</sub>. The extracts of either method were concentrated and purified using silica gel/alumina column purification to remove matrix interferences. The purified extract was analyzed for aromatic and chlorinated hydrocarbons by GC/MS or GC/ECD, respectively.

## 6. ANALYTICAL PROCEDURES FOR OTHER PARAMETERS

The NS&T Program quantifies additional parameters for all three matrices: fish, mollusks, and sediments. Parameters such as total organic carbon (TOC) can be used to "normalize" sediment contaminant values. Other parameters such as the quantification of fish bile for PAH metabolites, and fish, mollusks, and sediments for butyltins provide a more complete picture of contamination of the biotic environment. The amount of *Clostridium perfringens* spores in sediment is also quantified.

### 6.1. National Benthic Surveillance Project

#### 6.1.1. Sediment

Sediments are quantified for the same analytes as fish are, with the addition of PAHs. In addition, sediments are also characterized for the following ancillary parameters: TOC, total carbonate, percent dry weight, and the grain size distribution.

##### 6.1.1.1. Total organic carbon

Total organic carbon in sediments was determined instrumentally with a CHN analyzer on samples treated by the procedure of Hedges and Stern (1984). Total organic carbon (TOC) was measured directly in samples from which carbonate carbon was removed by acid volatilization. For high carbonate sediments, both TOC and total carbonate carbon were determined. Total carbonate was determined by the difference between treated and untreated sample aliquots. Determination of total organic carbon for the NBSP were performed by SEFSC.

##### 6.1.1.2. Moisture content

Sample moisture content was determined by weighing an undried sample, drying it either by freeze drying it or oven drying it at 120°C, and then reweighing the dry sample. If the sample was oven dried, it was allowed to cool in a dessicator before weighing.

##### 6.1.1.3. Particle size

Particle size determination on sediment samples differentiated the clay-silt component of the sediment (i.e., particles <63 μm in diameter) from the larger particles (Varanasi *et al.*, 1989). Sediments were separated into the two fractions using standard wet sieving techniques derived from EPA methods (Plumb, 1981). Determination of particle size for NBSP samples were performed by SEFSC.

#### 6.1.1.4. *Clostridium perfringens*

*Clostridium perfringens* a spore forming bacterium, is used as an indicator of sewage pollution in sediments. A discussion of its use and methods of quantification in the NS&T Program are found in Cabelli (1976), Emerson and Cabelli (1982), and Woodruff (1992). *Clostridium perfringens* colonies were enumerated from NBSP sediments from all coasts in 1984 by Cabelli (University of Rhode Island), in 1987-1988 by SEFSC, Charleston Laboratory, and in 1988-1992 by SEFSC, Beaufort Laboratory (P. Hanson, NOAA/NMFS/SEFSC, Beaufort, NC, personal communication, 1993).

#### 6.1.1.5. Coprostanol

Coprostanol was quantified for all NBSP sites in 1984 and 1987, using the method of MacLeod *et al.* (Volume IV, this document) and in the following years using the methods of Krahn *et al.*, 1989.

#### 6.1.2. Tissue

##### 6.1.2.1. Tissue dry weight

Fish livers and fish stomach contents were characterized for percent dry weight by weighing tissues before and after drying. Quantification of specific contaminants and tissue types are described below.

##### 6.1.2.2. Bile

Fish bile collection is discussed in the sampling protocols of the NBSP (Section 3.1.3) and in the NBSP organic analysis Section 5.4.2.

##### 6.1.2.3. Butyltins

Butyltin analyses for fish liver and sediments became a regular part of the NBSP in 1988. Analyses were performed on composites of 10 fish per site and on site sediment composites (3 grabs per station, 3 stations per site were composited into one butyltin sediment sample (Krone *et al.*, 1988a; Krone *et al.*, 1988b; and Krone *et al.*, 1991). Analyses were performed for tributyltin (TBT) and its metabolites (mono-, and di-butyltins), and tetrabutyltin. Results were reported as ng of total cation (mono-, di, tri-, tetra-butyltin).

The alkyltin chlorides were extracted from sediments or tissues using 0.1% tropolone in dichloromethane and anhydrous sodium sulfate. The tropolone is a complexing agent. The extracted alkyltins were converted into the n-hexyl derivatives using a Grignard reaction and silica/alumina column separation. For the liver extracts, an additional Sep-Pak separation was done prior to GC analysis by loading the appropriate silica/alumina column fraction on an amino Sep-Pak. The extracts were analyzed using GC/FID (Krone *et al.*, 1991).

##### 6.1.2.4. Otoliths

Otoliths or scales were used to estimate fish age. It is preferable to use otoliths of species that have calcareous otoliths since an additional layer of calcium is added approximately every year. Otoliths were used to determine the age of all the fish species collected in the NBSP with the exception of fourhorn sculpin, barred sand bass, and hornyhead turbot. Ages were determined for a representative sample of each fish length interval (Varanasi, 1989). These data are used to help interpret fish contaminant level and histopathology data.

## 6.2. Mussel Watch Project

### 6.2.1. Sediment

#### 6.2.1.1. Total organic and carbonate carbon

For samples collected on the Gulf Coast, carbon concentrations were determined on freeze dried or oven dried sediments. To determine total carbon, the sample was combusted with a carbon analyzer and the evolved CO<sub>2</sub> was analyzed by infrared detection. Total organic carbon was quantified by taking a dried sediment sample, removing the carbonate carbon with hydrochloric acid, and quantifying as above. Total carbonate carbon was calculated by the difference between total carbon and total organic carbon in samples (Wong *et al.*, this document).

The same method was used for samples collected on the East and West Coasts for analysis of total organic carbon. On the other hand, total inorganic carbon was quantified directly. Organic carbon was removed by heating and inorganic carbon was subsequently converted to CO<sub>2</sub> and quantified by measuring conductivity on a carbon analyzer (Padell and Hillman, this document).

#### 6.2.1.2. Moisture content

Sample moisture content was determined by weighing an undried sample, oven drying it and reweighing the dry sample. Samples were allowed to cool in a dessicator before reweighing. For East and West Coast samples, sediments were dried twice for 24 hr at 105°C with samples weighed after each drying (Padell and Hillman, this document). Gulf Coast samples were dried for 24 hr at 45°C and reweighed (Sweet *et al.*, this document).

#### 6.2.1.3. Particle size

Samples were collected in a plastic bag and refrigerated but not frozen. Hydrogen peroxide was added to approximately 15-20 g of homogenized sediment to oxidize organic matter present. The sample was washed with distilled water to remove salts, a solution of sodium hexametaphosphate was added to act as a dispersant and the mixture shaken for 24 hr. The sample was poured through a 62.5 µm screen to separate the gravel/sand fraction. The silt and clay fraction was collected and sizes quantified using the suspension, settling and pipetting techniques developed by Folk (1974). The sand and gravel fraction was oven dried and weighed. To differentiate between the sand and gravel, the coarse sediment was dry-sieved at 2 mm (-1.0 phi) and 62.5 micron (4 phi) intervals (GERG. 1990). These specific methods were used for the Gulf Coast portion of the MWP. Battelle used comparable methods to characterize grain size because their methods were also a modification of Folk (1974). SAIC, which characterized grain size for sediments samples from California and Hawaii in 1986 and 1987, followed methods of the American Society for Testing and Materials (ASTM, D422-58C; 1978 revision).

#### 6.2.1.4. *Clostridium perfringens*

*Clostridium perfringens* numbers have been quantified in the MWP since 1990. Methods used by both Battelle and GERG derive from work by Cabelli (1976) and Bisson and Cabelli (1979). Specific methods of both laboratories can be found in Volume II, this document.

#### 6.2.1.5. Coprostanol

Coprostanol was the indicator used to quantify the magnitude of human waste in the marine environment between 1986 and 1989. The method used to extract and quantify coprostanol for the MWP closely followed the method presented in MacLeod *et al.* (Volume IV, this document).

#### 6.2.2. Tissue

##### 6.2.2.1. Dry weight

Bivalve mollusk tissues were characterized for percent dry weight as were sediments. Approximately 500 mg of wet tissue was weighed, oven dried for 24 hr, allowed to cool, and reweighed. Procedures follow those used for sediments. Mussel Watch Project data are presented on a dry weight basis.

##### 6.2.2.2. Gonadal index

The degree of gonadal maturation or the degree to which gametes were spent was determined from mollusks taken from all MWP sites between 1986 and 1991, inclusive. The intent of this quantification was to ensure that primarily pre-spawning organisms were collected and analyzed for organic and inorganic contaminants. Because it was necessary to prepare slides to derive the degree of gonadal development, the opportunity arose to also measure the degree of parasitism and cell degeneration in mollusk samples (Hillman, 1991). Methods used to define the degree of gonadal development differ between the East and West, and the Gulf Coast portions of the project.

##### 6.2.2.2.1. East and West Coasts

The methods used by Battelle (1988), presented below, are similar to those developed by Seed (1975 and 1976). Most of the gonadal material of mussels and oysters is located in the mantle. Ordinary means of removal of tissues from shells usually results in severe damage to the mantle tissue which lies next to the shell. For that reason, it was necessary to exercise great care in shucking mollusk tissues to be used for gonadal index evaluation. After tissues were excised, they were preserved using a fixative solution.

Four stages in the reproductive cycle can be recognized in a histological section: developing, ripe, spawning, and spent. Developing and spawning stages can be further subdivided, resulting in a total of ten stages into one of which any individual can be assigned. Method specifics can be found in Hillman (Volume II, this document).

Each stage was assigned a number for 0 to 5 and for each bivalve site a mean population gonadal index was determined by multiplying the number of animals in each stage by the numerical ranking of the stage, and dividing the sum of those products by the total number of individuals in the sample.

The preparation of slides for the analysis of gonadal index also made it possible to examine bivalve tissues for histopathological conditions such as neoplasia. Slides were examined for the incidence of neoplasia on East and West Coast mollusk samples and correlations were made to organic contaminant concentrations (Hillman *et al.*, 1992).

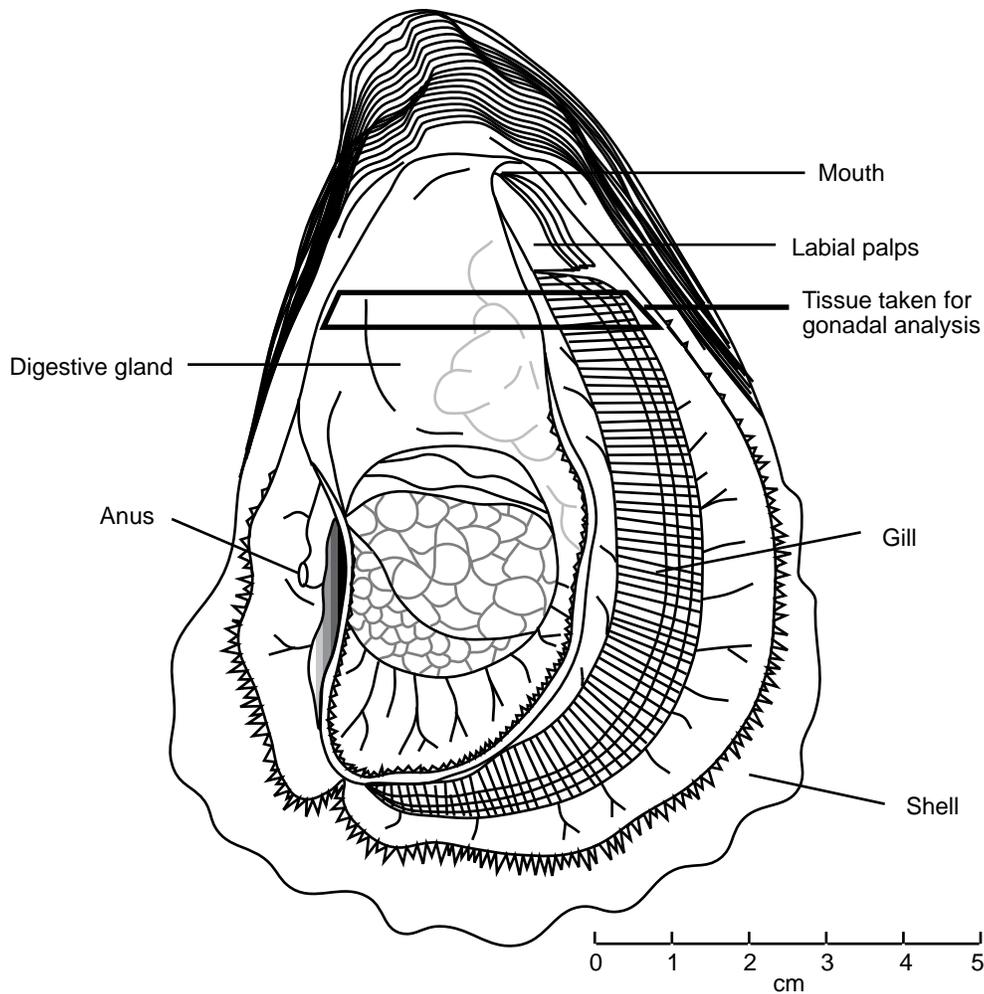


Figure I.15. Oyster tissue used for quantifying gonadal index (adapted from Galtsoff, 1964).

For the years 1986 through 1989, 10 bivalves were characterized for gonadal maturation from each of three stations per MWP site or for a total of 30 mollusks per site. For the years 1990 and 1991, fifteen mollusks were analyzed from each site. Gonadal indices remain fairly constant from year to year for the same site. Therefore, beginning in 1992, gonadal indices were no longer derived for all Project sites being sampled in a given year. This reduction in the number of gonadal analyses performed also applies to the Gulf Coast portion of the MWP.

#### 6.2.2.2.2. Gulf Coast

Methods used by GERG are provided below (GERG, 1990). Typically, Gulf Coast oysters are undifferentiated in the winter, the gonads begin to develop in early spring and spawning occurs during late spring through early fall. The state of gonadal development was determined by observation of histological section after Harris' hematoxylin and eosin staining. Oyster gender was determined and oysters were assigned to a semi-quantitative state of reproductive development.

A 5-mm thick cross section of tissue was removed from the oyster. The section was obtained such that the dorsal-ventral aspect pass through the digestive gland and gill tissue just

posterior to the palps, Figure 1.15. Each section was immediately placed in a tissue cassette and immersed in refrigerated Davidson's (or Bouin's fixative may be used as an alternative) for 48 hr. A slide was prepared by fixing a sample in paraffin. Tissue sections were sliced to 6  $\mu\text{m}$  on a microtome and affixed to slides using albumin adhesive. Staining was performed with Eosin Y, Orange G, and acid fuchsin stains.

Eight stages in the reproductive cycle can be recognized in a histological section: Sexually undifferentiated, early development, mid-development, late development, fully developed, spawning, spawned, spent. Method specifics can be found in Powell *et al.* (Volume II, this document). A quantitative method to define gonadal state, using an immunological probe, has been developed by Choi and Powell and is described in Volume II (this document).

While undergoing characterization for gonadal index, Gulf Coast oyster samples were also characterized for the degree of parasitism by *Bucephalus* sp., a trematode. (Brooks *et al.*, 1989).

#### 6.2.2.3. Butyltins

Thawed, anhydrous, homogenized tissue was extracted with troplone and hexane (East and West Coast samples) and troplone and dichloromethane (Gulf Coast samples) (Battelle, 1988; GERG, 1990). The extracts underwent Grignard reactions by addition of hexylmagnesium bromide (Gulf Coast samples) and n-pentyl magnesium bromide (East and West Coast samples). Florisil/silica gel or silica alumina column chromatography were used to separate the analytes. Quantitation of Sn was performed using GC/Flame Photometric Detection (FPD). Results were reported by Battelle as the cation (mono-, di-, tri-, tetra-butyltin) concentration (Uhler *et al.*, 1991; Volume IV, this document) and by TAMU on a  $\mu\text{g}$  Sn basis (GERG, 1992; Wade *et al.*, 1990).

## 7. GROSS PATHOLOGY

### 7.1. National Benthic Surveillance Project

Fish selected for chemical and histopathological analysis were also examined for gross pathology, particularly in the liver, and for fin erosion. Fin erosion was distinguished from damage caused by predatory animals and abrasions caused by the collecting net. It was characterized by changes including loss of tissue or fin rays, fusion of fin rays, and fin ray deformation (C. Stehr, NOAA/NMFS/NWFSC, Seattle, WA, personal communication, 1993).

### 7.2. Mussel Watch Project

Because of the soft nature of mollusk tissues and because of their protective shell covering, gross pathological conditions are usually not noted but in oysters from the Gulf of Mexico the parasitic trematode *Bucephalus* sp. was so prevalent that certain oysters had little or no gonadal tissue remaining (Brooks *et al.*, 1989).

## 8. HISTOPATHOLOGY

### 8.1. National Benthic Surveillance Project

Liver, kidney and gill sections from each of 60 individuals per site were excised and preserved in the field, and sectioned at five microns (Preece, 1972). When necessary, tissues such as gills and bones were decalcified using a commercial decalcification solution prior to processing.

Paraffin sections were stained with Mayer's hematoxylin and eosin. For further characterization of specific lesions, additional sections were stained observing standard staining methodologies (Preece, 1972; Armed Forces Institute of Pathology, 1968).

#### 8.1.1. Liver

All types of observed liver lesions were noted. Five classes of liver lesions that appear to be related to pollution exposure were examined in detail. These lesion types include neoplasms, foci of cellular alteration (putative preneoplastic lesions), specific degeneration/necrosis, proliferative lesions, and hydropic vacuolation (also call atypical vacuolated cells or "RAM" cells) (B. McCain, NOAA/NMFS/NWFSC, Seattle, WA, personal communication, 1992). Further information discussing this topic can be found in Varanasi *et al.*, 1988.

#### 8.1.2. Kidneys

Three categories of kidney lesions which appeared to be pollution-related were also examined in more detail. These include kidney necrosis, proliferation, and sclerosis (B. McCain, NOAA/NMFS/NWFSC, Seattle, WA, personal communication, 1992).

#### 8.1.3. Gills

Gills were examined for lesions through 1987. Five types of conditions were documented: Degenerative/necrotic lesions including respiratory epithelial necrosis, hypertrophy, and hydropic degeneration; proliferative lesions, including respiratory epithelial hyperplasia, lamellar fusion, filament epithelial hyperplasia, pillar cell proliferation, and mucous cell hyperplasia; vascular lesions, including microaneurysms or telangiectasia, and intravascular thrombi; inflammatory lesions, including lymphoid infiltrates, edema, and chronic inflammation; and depositional disorders, represented by thickening of the lamellar base membrane.

Although lesions were seen in gills, there were no significant intersite differences in prevalences, there was no relationship to contaminant exposure, and the lesions were nonspecific. For these reasons, gills were not examined for histopathological disorders after 1987 ( Mark Myers, NOAA/NMFS/NWFSC, Seattle, WA, personal communication, June 1993).

### 8.2. Mussel Watch Project

The main incidence of mollusk disease documented by MWP for the East and West Coasts was neoplasia (Battelle, 1991; Hillman *et al.*, 1992). The microsporan *Steinhausia mytilovum* was documented for the first time in *Mytilus* sp. from California (Hillman, 1991).

Infection with *Perkinsus marinus* is the most common cause of mortality in Gulf Coast oysters (Powell *et al.*, 1992). A tissue homogenate or a section of mantle tissue is incubated in thioglycollate medium for 14 days according to the method of Ray (1966). A semiquantitative (Craig *et al.*, 1989) or quantitative (Choi *et al.*, in press) assessment of hypnospore number is then made microscopically (Ormond-Wilson *et al.*, Volume II, this document). TAMU GERG has been quantifying the incidence of this protozoan since 1986.

## 9. SUMMARY

Over the years, NOAA's NS&T Program has evolved in response to new information and better analytical methods, while retaining its core of sites and analytes to allow examination of long-term trends. Success of NOAA's monitoring program requires flexibility in introduction of

newer technologies, in expansion of the number of matrices that are monitored, in the quantification of ancillary parameters, inclusion of new environmental contaminants, retention of a specimen bank and a strong QA/QC Project. NOAA's program encourages flexibility yet retains consistency. The MWP has seven years of monitoring data with the eighth year already underway. The NBSP has the seventh year of data processed with the tenth year of monitoring underway. This extensive database will allow the environmental community to evaluate the success of recent attempts to improve environmental quality.

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## Appendix A

### Background Information on the NS&T Program

Table A.1. Recommended detection limits for core target chemicals ( $\mu\text{g/g}$ ) (Boehm, 1983).

Organics <sup>Δ</sup>	Recommended detection limits		Metal <sup>◇</sup>	Recommended detection limits	
	Sediments	Tissues		Sediments	Tissues
PAH (individual compounds)	0.0010	0.010	Hg	0.005	0.001
PCB (individual congeners)	0.0001	0.001	Cd	0.005	0.001
Pesticides (individual compounds)	0.0001	0.001	Pb	0.2	0.04
			As	0.05	0.01
			Cu	0.05	0.01
			Cr	0.2	0.04
			Ag	0.005	0.001

<sup>Δ</sup> Based on 100 g of wet sediment sample or 50 g dry weight (concentrations reported on a dry weight basis); based on 100 g of wet tissue (concentrations reported on a wet weight basis).

<sup>◇</sup> Based on approximately 0.5 grams of dry sediment (concentrations reported in a dry weight basis); based on approximately 0.5 g of dry tissue, approximately 2.5 g wet tissue (concentrations reported on a wet weight basis).

Table A.2. Minimum technical expertise required for trace and major element analyses of bivalve tissues and surface sediments ( $\mu\text{g/g}$  dry weight).\*

Element	Tissues	Sediments	Elements	Tissues	Sediments
Al	10.0	1500	As	2.0	1.5
Si	100	10000	Se	1.0	0.1
Cr	0.1	5.0	Ag	0.01	0.01
Mn	5.0	1.0	Cd	0.2	0.05
Fe	50.0	500	Sn	0.05	0.1
Ni	0.5	1.0	Sb	0.2	0.2
Cu	5.0	5.0	Hg	0.01	0.01
Zn	50.0	2.0	Pb	0.1	1.0

\* For the Mussel Watch Project years 1990-1994 NOAA defined at what level contaminants should be quantified. These levels were not defined as detection limits because actual detection limits are a function of a specific sample size, and other analytical variables.

Table A.3. Minimum technical expertise required for polynuclear aromatic hydrocarbon determinations (ng/g dry weight).\*

Compound	Tissues	Sediments	Compound	Tissues	Sediments
Naphthalene	20	5	Anthracene	20	5
2-Methylnaphthalene	20	5	1-Methylphenanthrene	20	5
1-Methylnaphthalene	20	5	Fluoranthene	20	5
Biphenyl	20	5	Pyrene	20	5
2,6-Dimethylnaphthalene	20	5	Chrysene	20	5
Acenaphthene	20	5	Benz[ <i>a</i> ]anthracene	20	5
Acenaphthylene	20	5	Benzo[ <i>b</i> ]fluoranthene	20	5
1,6,7-Trimethylnaphthalene	20	5	Benzo[ <i>k</i> ]fluoranthene	20	5
Fluorene	20	5	Benzo[ <i>ghi</i> ]perylene	20	5
Dibenz[ <i>a,h</i> ]anthracene	20	5	Benzo[ <i>e</i> ]pyrene	20	5
Indeno[1,2,3- <i>cd</i> ]pyrene	20	5	Benzo[ <i>a</i> ]pyrene	20	5
Phenanthrene	20	5	Perylene	20	5

\* For the Mussel Watch Project years 1990-1994 NOAA defined at what level contaminants should be quantified. These levels were not defined as detection limits because actual detection limits are a function of a specific sample size, and other analytical variables.

Table A.4. Minimum technical expertise required for pesticides and PCBs determinations (ng/g dry weight).\*

Compound	Tissues	Sediments	Compound	Tissues	Sediments
Aldrin	0.25	0.1	PCB 8	1.0	0.1
<i>cis</i> -Chlordane	0.25	0.1	PCB 18	1.0	0.1
Dieldrin	0.25	0.1	PCB 28	1.0	0.1
Heptachlor	0.25	0.1	PCB 44	1.0	0.1
Heptachlor epoxide	0.25	0.1	PCB 52	1.0	0.1
Hexachlorobenzene	0.25	0.1	PCB 66	1.0	0.1
gamma-HCH	0.25	0.1	PCB 77/110	1.0	0.1
Mirex	0.25	0.1	PCB 101	1.0	0.1
<i>trans</i> -Nonachlor	0.25	0.1	PCB 118	1.0	0.1
Endrin	0.25	0.1	PCB 126	1.0	0.1
			PCB 128	1.0	0.1
			PCB 128	1.0	0.1
2,4'-DDD	0.25	0.1	PCB 138	1.0	0.1
4,4'-DDD	0.25	0.1	PCB 153	1.0	0.1
2,4'-DDE	0.25	0.1	PCB 170	1.0	0.1
4,4'-DDE	0.25	0.1	PCB 180	1.0	0.1
2,4'-DDT	0.25	0.1	PCB 187	1.0	0.1
4,4'-DDT	0.25	0.1	PCB 195	1.0	0.1
			PCB 206	1.0	0.1
			PCB 209	1.0	0.1

\* For the Mussel Watch Project years 1990-1994 NOAA defined at what level contaminants should be quantified. These levels were not defined as detection limits because actual detection limits are a function of a specific sample size, and other analytical variables.

Table A.5. National Benthic Surveillance Project Northeast Coast sediment aromatic hydrocarbons, lowest reported concentrations (ng/g dry weight).

Compound	1984	1985	1986	1987	1988
1-Methylnaphthalene	15	0.5	1.6	2.0	0.9
1-Methylphenanthrene	3.0	3.0	1.8	2.0	0.7
2,6-Dimethylnaphthalene	3.4	12	0.89	3.0	0.9
2-Methylnaphthalene	12	1.6	2.0	3.0	2.0
Acenaphthene	6.6	4.4	4.6	3.0	0.7
Anthracene	7.3	3.6	3.8	0.6	0.7
Benzo[ <i>a</i> ]anthracene	4.0	3.6	5.0	3.0	4.0
Benzo[ <i>a</i> ]pyrene	3.1	1.0	1.7	3.0	5.0
Benzo[ <i>e</i> ]pyrene	2.3	0.5	5.6	2.0	6.0
Biphenyl	1.3	6.7	4.0	2.0	0.6
Chrysene	7.4	2.6	0.77	5.0	0.5
Dibenz[ <i>a,h</i> ]anthracene	13	0.4	14	3.0	0.6
Fluoranthene	3.0	1.3	3.1	4.0	2.0
Fluorene	5.7	5.0	3.9	2.0	1.0
Naphthalene	7.2	6.6	2.2	2.0	1.0
Perylene	0.93	0.7	7.6	3.0	2.0
Phenanthrene	6.0	3.3	3.9	6.0	3.0
Pyrene	8.7	1.8	3.8	5.0	2.0

Table A.6. National Benthic Surveillance Project Northeast Coast tissue chlorinated pesticides, lowest reported concentrations (ng/g dry weight).

Compound	1984	1985	1986
Aldrin	11	6.0	4.7
<i>cis</i> -chlordane	18	9.0	4.7
Dieldrin	11	8.0	11
Heptachlor	2.6	1.0	1.1
Heptachlor epoxide	2.6	4.0	2.5
Hexachlorobenzene	2.4	1.0	1.1
Lindane	1.7	1.0	1.1
Mirex	1.3	1.0	1.9
<i>trans</i> -Nonachlor	8.3	10	24
2,4'-DDE	9.9	11	3.8
4,4'-DDE	11	18	82
2,4'-DDD	3.8	3.0	2.8
4,4'-DDD	6.1	6.0	11
2,4'-DDT	4.8	4.0	2.4
4,4'-DDT	7.4	2.0	5.8

Table A.7. National Benthic Surveillance Project Northeast Coast sediment chlorinated pesticides, lowest reported concentrations (ng/g dry weight).

Compound	1984	1985	1986	1987	1988
Aldrin	0.78	0.06	0.02	-	-
<i>cis</i> -chlordane	0.35	0.1	0.09	0.5	0.2
Dieldrin	0.62	0.2	0.64	0.9	0.3
Heptachlor	0.37	0.4	0.13	2.0	0.9
Heptachlor epoxide	-	0.2	0.36	0.4	0.3
Hexachlorobenzene	0.1	0.1	0.12	0.5	0.4
gamma-HCH	0.23	0.04	0.15	0.5	0.5
Mirex	0.5	0.4	1.0	0.2	0.3
<i>trans</i> -Nonachlor	0.2	0.2	0.19	0.4	1.0
2,4'-DDE	0.4	0.8	0.61	0.7	0.8
4,4'-DDE	0.16	0.2	0.24	0.4	0.4
2,4'-DDD	1.2	0.1	0.81	0.4	0.5
4,4'-DDD	0.41	0.1	0.21	0.5	0.8
2,4'-DDT	1.8	0.1	0.36	-	-
4,4'-DDT	0.09	0.05	0.11	0.8	0.6

Table A.8. National Benthic Surveillance Project Northeast Coast tissue polychlorinated biphenyls, lowest reported concentrations (ng/g dry weight).

Compound	1984	1985	1986
Dichlorobiphenyls	6.2	10	2.1
Trichlorobiphenyls	1.5	8.0	14
Tetrachlorobiphenyls	12	37	28
Pentachlorobiphenyls	49	44	44
Hexachlorobiphenyls	130	110	84
Heptachlorobiphenyls	31	68	60
Octachlorobiphenyls	3.4	3.0	16
Nonachlorobiphenyls	10	4.0	2.5

Table A.9. National Benthic Surveillance Project Southeast and Gulf Coasts sediment aromatic hydrocarbons, lowest reported concentrations (ng/g dry weight).

Compound	1984	1985	1986	1987	1988
1-Methylnaphthalene	4.3	2.6	-	140	3.0
1-Methylphenanthrene	5.2	3.6	86	25	2.0
1,6,7-Trimethylnaphthalene	-	-	4.5	4.8	0.6
2,6-Dimethylnaphthalene	61	3.4	700	64	3.0
2-Methylnaphthalene	7.0	6.9	83	43	2.0
Acenaphthene	100	2.6	3.8	1.5	2.0
Acenaphthylene	-	-	13	7.4	1.0
Anthracene	4.6	6.7	220	44	3.0
Benzofluoranthene	-	-	-	-	-
Benzo[ <i>g,h,i</i> ]perylene	-	-	100	32	5.0
Benzo[ <i>a</i> ]anthracene	11	5.2	120	20	3.0
Benzo[ <i>a</i> ]pyrene	1.9	6.0	120	35	5.0
Benzo[ <i>e</i> ]pyrene	3.3	2.7	260	70	7.0
Biphenyl	7.2	5.6	28	6.7	3.0
Chrysene	5.5	6.7	190	80	3.0
Dibenz[ <i>a,h</i> ]anthracene	5.3	3.6	210	57	0.9
Fluoranthene	5.2	7.4	180	66	4.0
Fluorene	1.6	6.3	23	4.8	1.0
Indeno[1,2,3- <i>cd</i> ]pyrene	-	-	110	62	3.0
Naphthalene	5.1	4.0	300	71	3.0
Perylene	4.6	2.6	-	110	3.0
Phenanthrene	6.9	6.9	240	87	2.0
Pyrene	2.7	5.3	270	69	3.0

Table A10. National Benthic Surveillance Project Southeast and Gulf Coasts sediment pesticides, lowest reported concentrations (ng/g dry weight).

Aldrin	-	-	-	-	-
<i>cis</i> -chlordane	0.74	0.77	-	1.5	0.4
Dieldrin	1.1	1.3	-	1.4	0.1
Heptachlor	-	1.3	-	-	1.0
Heptachlor epoxide	-	-	-	-	0.1
Hexachlorobenzene	0.08	1.9	-	1.6	0.1
gamma-HCH	-	0.52	-	1.3	0.6
Mirex	0.92	-	-	-	-
<i>trans</i> -Nonachlor	0.42	-	-	0.7	0.5
2,4'-DDE	0.89	0.84	-	-	2.0
4,4'-DDE	0.06	1.2	1.2	1.0	0.4
2,4'-DDD	0.79	0.56	-	1.2	0.4
4,4'-DDD	1.9	0.56	3.9	3.3	0.8
2,4'-DDT	-	-	-	2.2	-
4,4'-DDT	2.0	0.53	-	5.0	1.0

Table A.11. National Benthic Surveillance Project Southeast and Gulf Coasts sediment polychlorinated biphenyls, lowest reported concentrations (ng/g dry weight).

Compound	1984	1985	1986	1987	1988
Dichlorobiphenyls	11	1.0	-	-	-
Trichlorobiphenyls	0.33	0.52	-	-	0.3
Tetrachlorobiphenyls	0.72	0.59	-	-	2.0
Pentachlorobiphenyls	1.0	0.52	19	-	0.4
Hexachlorobiphenyls	2.1	2.0	-	-	0.1
Heptachlorobiphenyls	0.99	2.4	-	-	0.2
Octachlorobiphenyls	0.06	0.7	-	-	0.09
Nonachlorobiphenyls	1.6	1.2	-	-	2.0

Compound	1986	1987	1988	Compound	1986	1987	1988
PCB 8	-	-	-	PCB 128	-	1.9	0.3
PCB 18	-	-	2.0	PCB 138	-	6.1	0.1
PCB 28	-	-	0.4	PCB 153	2.9	5.4	0.1
PCB 44	1.5	1.2	0.3	PCB 170	-	3.5	0.2
PCB 52	2.5	2.7	0.4	PCB 180	1.8	1.9	0.8
PCB 66	-	3.1	0.4	PCB 187	1.4	1.9	0.8
PCB 101	3.3	1.6	0.2	PCB 195	-	1.1	0.2
PCB 105	1.9	1.2	0.07	PCB 206	-	1.6	2.0
PCB 118	3.5	2.4	0.1	PCB 209	-	1.9	0.3

Table A.12. National Benthic Surveillance Project Northeast Coast sediment polychlorinated biphenyls, lowest reported concentrations (ng/g dry weight).

Compound	1986	1987	1988	Compound	1986	1987	1988
PCB 8	0.55	-	-	PCB 128	0.29	0.3	0.3
PCB 18	0.34	1.0	4.0	PCB 138	0.14	0.5	1.0
PCB 28	0.12	1.0	0.6	PCB 153	0.11	0.4	0.6
PCB 44	0.59	1.0	0.5	PCB 170	0.13	0.3	0.3
PCB 52	0.29	2.0	0.7	PCB 180	0.12	0.4	0.4
PCB 66	0.17	0.5	1.0	PCB 187	0.18	0.6	0.5
PCB 101	0.08	0.9	1.0	PCB 195	0.08	0.3	0.7
PCB 105	0.29	0.3	0.4	PCB 206	0.11	0.2	0.3
PCB 118	0.08	0.9	0.6	PCB 209	0.11	0.7	0.2
Dichlorobiphenyls		1.5	1.0	0.6	-	-	
Trichlorobiphenyls		0.61	0.6	0.15	2.0	1.0	
Tetrachlorobiphenyls		0.42	0.2	0.09	2.0	3.0	
Pentachlorobiphenyls		0.2	0.2	0.21	2.0	1.0	
Hexachlorobiphenyls		0.2	0.5	0.14	0.4	0.7	
Heptachlorobiphenyls		0.2	0.2	0.13	2.0	0.6	
Octachlorobiphenyls		0.19	0.1	0.34	0.2	0.6	
Nonachlorobiphenyls		0.37	0.3	0.11	0.2	0.3	

Table A.13. National Benthic Surveillance Project Southeast and Gulf Coasts tissue chlorinated pesticides and PCBs, lowest reported concentrations (ng/g dry weight).

Compound	1984	1985	1986	1987
Aldrin	3.9	-	-	-
<i>cis</i> -chlordane	3.1	5.0	7.3	7.0
Dieldrin	3.6	4.9	7.6	11.0
Heptachlor	3.8	-	-	-
Heptachlor epoxide	3.5	3.7	4.9	6.0
Hexachlorobenzene	5.9	3.0	4.5	5.0
Lindane	3.3	4.2	4.6	8.0
Mirex	3.7	3.0	4.2	3.0
<i>trans</i> -Nonachlor	3.1	3.2	5.9	7.0
2,4'-DDE	4.0	3.3	110	17
4,4'-DDE	5.1	4.5	12	11
2,4'-DDD	3.7	3.3	4.4	3.0
4,4'-DDD	5.2	4.2	10	21
2,4'-DDT	3.3	3	3.9	10
4,4'-DDT	3.7	4.4	7.3	6.0
Dichlorobiphenyls	7.2	7.1	4.6	-
Trichlorobiphenyls	3.1	4.0	24	52
Tetrachlorobiphenyls	4.7	12	84	190
Pentachlorobiphenyls	3.2	4.2	86	280
Hexachlorobiphenyls	3.5	7.2	120	170
Heptachlorobiphenyls	3.9	4.3	66	110
Octachlorobiphenyls	4.3	3.2	29	12
Nonachlorobiphenyls	3.7	3.0	4.8	22

Table A.14. National Benthic Surveillance Project West Coast sediment aromatic hydrocarbons, lowest reported concentrations (ng/g dry weight).\*

Compound	1984	1985	1986	1987	1988
1-Methylnaphthalene	5.0	1.5	3.5	1.0	2.0
1-Methylphenanthrene	6.0	3.0	3.0	0.9	1.0
1,6,7-Trimethylnaphthalene	-	-	18	1.0	2.0
2,6-Dimethylnaphthalene	1.0	1.5	10	2.0	3.0
2-Methylnaphthalene	6.0	5.0	6.0	2.0	2.0
Acenaphthene	0.9	6.0	4.5	0.4	3.0
Acenaphthylene	-	-	5.0	1.0	-
Anthracene	4.0	3.0	5.0	3.0	2.0
Benzo[fluoranthene	-	-	4.0	0.6	-
Benzo[ <i>b</i> ]fluoranthene	-	-	-	-	-
Benzo[ <i>k</i> ]fluoranthene	-	-	-	-	-
Benzo[ <i>ghi</i> ]perylene	-	-	5.0	0.9	9.0
Benzo[ <i>a</i> ]anthracene	6.0	2.0	2.0	0.5	2.0
Benzo[ <i>a</i> ]pyrene	6.0	7.0	13	1.0	1.0
Benzo[ <i>e</i> ]pyrene	3.5	5.5	5.0	0.6	0.6
Biphenyl	1.0	5.0	4.0	1.0	1.0
Chrysene	4.0	3.0	9.0	2.0	6.0
Dibenz[ <i>a,h</i> ]anthracene	7.0	6.5	-	2.0	0.8
Fluoranthene	12	2.0	4.0	0.7	1.0
Fluorene	2.0	3.5	4.0	1.0	0.7
Indeno[1,2,3- <i>cd</i> ]pyrene	-	-	14	14	1.0
Naphthalene	7.0	1.5	6.0	1.0	1.0
Perylene	11	3.0	5.0	0.6	3.0
Phenanthrene	3.5	4.5	5.0	0.4	0.4
Pyrene	3.5	1.5	3.0	0.9	0.9

\* See footnote page 80.

Table A.15. National Benthic Surveillance Project West Coast sediment chlorinated pesticides, lowest reported concentrations (ng/g dry weight).\*

Aldrin	-	-	2.0	-	-
<i>cis</i> -chlordane	0.35	0.2	0.6	0.2	0.3
Dieldrin	2.0	0.15	0.7	0.3	0.3
Heptachlor	-	0.3	0.7	0.3	0.1
Heptachlor epoxide	-	0.8	0.45	0.4	0.7
Hexachlorobenzene	0.4	0.05	0.4	0.4	0.3
gamma-HCH	-	0.05	0.5	0.3	0.9
Mirex	2.0	0.7	0.35	0.3	0.4
<i>trans</i> -Nonachlor	0.2	0.1	0.4	0.3	0.2
2,4'-DDE	0.5	0.1	1.0	0.5	0.3
4,4'-DDE	0.1	0.1	0.5	0.4	0.3
2,4'-DDD	0.3	0.2	1.0	0.4	0.3
4,4'-DDD	0.2	0.2	1.0	0.3	0.5
2,4'-DDT	-	-	-	-	-
4,4'-DDT	0.6	0.15	0.7	0.3	0.8

Table A.16. National Benthic Surveillance Project West Coast sediment polychlorinated biphenyls, lowest reported concentrations (ng/g dry weight).\*

Compound	1984	1985	1986	1987	1988	
Dichlorobiphenyls	3.0	1.0	-	-	-	
Trichlorobiphenyls	0.2	0.4	0.5	0.9	0.3	
Tetrachlorobiphenyls	0.3	0.2	1.0	3.0	0.8	
Pentachlorobiphenyls	0.6	0.6	0.5	0.6	0.6	
Hexachlorobiphenyls	0.2	0.1	1.0	0.5	0.2	
Heptachlorobiphenyls	0.5	0.15	0.9	0.6	0.1	
Octachlorobiphenyls	0.4	0.03	0.5	0.5	0.1	
Nonachlorobiphenyls	0.1	0.3	0.25	0.2	0.2	
Decachlorobiphenyl	-	-	-	-	-	
PCB 8	-	-	-	0.9	0.6	0.09
PCB 18	2	1	0.5	0.4	0.4	0.2
PCB 28	2	0.6	0.3	0.6	0.5	0.2
PCB 44	1	1	0.6	0.6	1	1
PCB 52	1	1	0.6	0.6	0.6	0.5
PCB 66	0.5	1	0.6	1	0.4	0.1
PCB 101	0.6	0.2	0.2	0.5	0.3	0.1
PCB 105	0.6	0.2	0.2	0.35	0.2	0.2
PCB 118	0.4	0.2	0.3	0.2	0.5	0.4

\* See footnote p. 80.

Table A.17. National Benthic Surveillance Project West Coast tissue chlorinated pesticides and PCBs, lowest reported concentrations (ng/g dry weight).\*

Compound	1984	1985	1986	1987	1988
Aldrin	-	1.0	5.0	-	-
<i>cis</i> -chlordane	4.0	3.0	5.0	2.0	2.0
Dieldrin	4.0	3.0	2.0	2.0	1.0
Heptachlor	-	1.0	1.0	1.0	1.0
Heptachlor epoxide	-	1.0	2.0	1.0	1.0
Hexachlorobenzene	5.0	1.0	1.0	3.0	1.0
Lindane	7.0	1.0	1.0	2.0	1.0
Mirex	2.0	1.0	1.0	2.0	1.0
<i>trans</i> -Nonachlor	7.0	4.0	7.0	5.0	1.0
2,4'-DDE	3.0	3.0	5.0	3.0	1.0
4,4'-DDE	10	3.0	14	2.0	12
2,4'-DDD	3.0	2.5	2.0	2.0	2.0
4,4'-DDD	9.0	7.0	6.0	2.0	3.0
2,4'-DDT	-	-	-	-	-
4,4'-DDT	5.0	1.0	2.0	1.0	1.0
Dichlorobiphenyls	3.0	3.0	-	-	-
Trichlorobiphenyls	4.0	3.0	5.0	5.0	5.0
Tetrachlorobiphenyls	23	11	8.0	29	50
Pentachlorobiphenyls	76	7.0	26	58	73
Hexachlorobiphenyls	60	6.0	36	33	27
Heptachlorobiphenyls	22	20	14	16	13
Octachlorobiphenyls	4.0	2.0	2.0	12	2.0
Nonachlorobiphenyls	2.0	2.0	1.0	2.0	2.0
Decachlorobiphenyl	-	-	-	-	-

\* The actual detection limit for an individual analyte in a sample depends on factors such as the procedure used to analyze the sample, the sample weight, the percent dry weight, the smallest GC peak area of any detected analyte in the appropriate GC calibration solution with the lowest concentration analyzed with the sample, and the GC detector response to the individual analyte relative to the GC internal standard. Approximate 1993 detection limits for NBSP sediments based on a 10 g sample size and a 60% dry weight are 0.2 to <2 ng/g for chlorinated hydrocarbons and 2 to <8 ng/g for PAHs. The approximate 1993 detection limits for livers based on a 3 g sample size and a 30% dry weight are 0.5 to <5 ng/g for chlorinated hydrocarbons. Stomach contents detection limits for a sample of 3 g and 20% dry weight were 0.5 to <5 µg/g for chlorinated hydrocarbons and 0.3 to <2 ng/g for aromatic hydrocarbons; C. Sloan, NOAA/NMFS/NWFSC, Seattle, WA, personal communication, 1993.

Table A.18. National Benthic Surveillance Project Northeast Coast sediment major and trace elements, detection limits and lowest reported concentrations ( $\mu\text{g/g}$  dry weight).

Elements	1984-1986*	1987 $\Delta$	1988-1993 $\dagger$
Al	2000	-	900
Si	20000	18	8000
Cr	6	43	2
Mn	3	410	20
Fe	2000	100	500
Ni	0.3	14	2
Cu	4	5.32	0.8
Zn	4	44	5
As	0.1	4.9	0.8
Se	0.1	-	0.05
Ag	0.01	0.03	0.02
Cd	0.01	0.07	0.02
Sn	0.2	2.7	0.3
Sb	0.2	0.31	0.8
Hg	0.01	-	0.06
Tl	0.2	-	0.3
Pb	0.1	15	0.5

\* Typical detection limits, taken from Zdanowicz *et al.*, this document.

$\Delta$  Lowest reported values.

$\dagger$  Typical detection limits, taken from Evans and Hanson, this document.

Table A.19. National Benthic Surveillance Project Northeast Coast tissue major and trace elements, detection limits and lowest reported concentrations ( $\mu\text{g/g}$  dry weight).

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Elements	1984-1986*	1987 $\Delta$	1988-1993 $\dagger$
Al	0.1	-	-
Si		-	-
Cr	0.04	0.36	0.05
Mn	1	2.1	1
Fe	5	0.01	5
Ni	0.1	0.05	0.1
Cu	2	1.8	1
Zn	1	59	2
As	0.1	1.3	0.3
Se	0.2	3.3	0.4
Ag	0.01	0.05	0.02
Cd	0.02	0.02	0.01
Sn	0.2	0.01	0.2
Sb	0.2	-	0.2
Hg	0.01	0.08	0.05
Tl	0.2	-	0.1
Pb	0.05	0.2	0.1

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\* Typical detection limits, taken from Zdanowicz *et al.*, this document.

$\Delta$  Lowest reported values.

$\dagger$  Typical detection limits, taken from Evans and Hanson, this document.

Table A.20. National Benthic Surveillance Project Southeast and Gulf Coasts sediment major and trace elements, detection limits ( $\mu\text{g/g}$  dry weight).<sup>†</sup>

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Elements	1984-1993
Al	900
Si	8000
Cr	2
Mn	20
Fe	500
Ni	2
Cu	0.8
Zn	5
As	0.8
Se	0.05
Ag	0.02
Cd	0.02
Sn	0.3
Sb	0.8
Hg	0.06
Tl	0.3
Pb	0.5

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<sup>†</sup> Typical detection limits, taken from Evans and Hanson, this document.

Table A.21. National Benthic Surveillance Project Southeast and Gulf Coasts tissue major and trace elements, detection limits ( $\mu\text{g/g}$  dry weight).<sup>†</sup>

Elements	1984-1933
Al	-
Si	-
Cr	0.05
Mn	1
Fe	5
Ni	0.1
Cu	1
Zn	2
As	0.3
Se	0.4
Ag	0.02
Cd	0.01
Sn	0.2
Sb	0.2
Hg	0.05
Tl	0.1
Pb	0.1

<sup>†</sup> Typical detection limits, taken from Evans and Hanson, this document.

Table A.22. National Benthic Surveillance Project West Coast sediment major and trace elements, lowest reported concentrations ( $\mu\text{g/g}$  dry weight).

Elements	1984	1985	1986	1987	1988
Al	22000	24000	28000	27000	44000
Si	160000	210000	220000	220000	170000
Cr	21	29	15	26	27
Mn	140	130	49	29	190
Fe	5000	7100	5500	3500	15000
Ni	2.0	2.9	1.9	4.81	4.1
Cu	4.3	1.2	3.6	2.0	0.55
Zn	12	15	5.6	8.1	38
As	0.35	0.52	0.38	3.4	1.5
Se	0.05	0.06	0.02	0.02	0.02
Ag	0.02	0.03	0.01	0.02	0.06
Cd	0.01	0.14	0.08	0.01	0.02
Sn	0.21	0.25	1.1	1.1	0.8
Sb	0.28	0.25	0.01	0.15	0.29
Hg	0.03	0.02	0.01	0.04	0.01
Tl	0.04	0.14	-	-	-
Pb	0.99	2.1	7.8	2.8	0.3

Table A.23. National Benthic Surveillance Project West Coast tissue major and trace elements, lowest reported concentrations ( $\mu\text{g/g}$  dry weight).

Elements	1984	1985	1986	1987
Al	-	-	-	-
Si	-	-	-	-
Cr	0.04	0.02	0.02	0.09
Mn	1.9	1.0	0.37	0.16
Fe	52	28	10	0.001
Ni	0.11	0.1	0.05	0.05
Cu	3.7	1.3	1.7	3.0
Zn	55	47	44.8	33
As	0.38	0.98	2.7	3.3
Se	0.64	2.5	1.8	0.81
Ag	0.04	0.02	0.01	0.01
Cd	0.32	0.24	0.01	0.08
Sn	0.14	0.1	0.18	0.01
Sb	-	-	-	-
Hg	0.11	0.03	0.03	0.02
Tl	-	-	-	-
Pb	0.07	0.04	0.02	0.03

Table A.24. Mussel Watch Project East and Northwest Coasts sediment polycyclic aromatic hydrocarbons, method limits of detection (ng/g dry weight).<sup>Δ</sup>

Compound	1986▼	1987◆	1988■	1989▲	1990*	1991-1992†
Acenaphthene	2.0	12	12	0.28	1.0	1.1
Acenaphthylene	-	-	9.7	2.0	2.3	1.4
Anthracene	2.7	13	13	1.3	3.2	1.3
Benz[ <i>a</i> ]anthracene	4.0	6.3	6.3	1.7	3.8	1.5
Dibenz[ <i>a,h</i> ]anthracene	2.3	22	23	1.0	0.74	1.4
Benzo[ <i>b</i> ]fluoranthene	-	-	4.5	2.9	3.4	1.4
Benzo[ <i>k</i> ]fluoranthene	-	-	3.7	1.1	3.0	1.7
Benzo[ <i>ghi</i> ]perylene	-	-	8.0	1.1	3.0	2.6
Benzo[ <i>a</i> ]pyrene	1.4	7.5	7.5	0.67	3.3	0.99
Benzo[ <i>e</i> ]pyrene	1.4	3.8	3.8	0.84	2.2	1.5
Biphenyl	0.81	11	11	0.66	1.9	1.2
Chrysene	10	8.7	8.7	0.56	5.4	12
2,6-Dimethylnaphthalene	2.3	14	14	1.1	1.8	1.2
Fluoranthene	4.5	11	11	1.5	8.0	2.7
Fluorene	0.81	7.2	7.2	0.51	0.70	0.83
Indeno[1,2,3- <i>cd</i> ]pyrene	-	-	17	1.51	2.7	2.4
1-Methylnaphthalene	5.7	14	14	0.63	1.9	0.86
2-Methylnaphthalene	2.3	13	13	0.72	1.9	0.87
1-Methylphenanthrene	0.81	8.1	8.1	1.3	2.1	1.7
Naphthalene	3.6	5.4	5.4	0.47	1.5	0.48
Perylene	1.6	6.1	6.1	0.39	2.7	4.6
Phenanthrene	2.3	9.9	9.9	0.92	5.0	1.4
Pyrene	4.2	28	28	1.4	7.6	2.4
1,6,7-Trimethylnaphthalene	-	16	16	0.79	1.1	1.4

<sup>Δ</sup> For 1990 and afterwards, detection limits are applicable to the East and the entire West Coasts.

▼ Boehm *et al.* (1987); ◆ Boehm *et al.* (1988); ■ Freitas *et al.* (1989); ▲ Battelle (1990b); \* Battelle, 1991; † Battelle 1992.

Table A.25. Mussel Watch Project East Coast additional oyster pesticides, method limits of detection (ng/g dry weight).

Compound	1989 <sup>▲</sup>
Technical Toxaphene	4.2
Endosulfan I	0.30
Endosulfan II	0.38
Atrazine	0.58
Propanil	9.5
Methyl Parathion	0.98
Carbaryl	6.6
Alachlor	3.1

<sup>▲</sup> Battelle, 1990b.

Table A.26. Mussel Watch Project East and Northwest Coasts sediment chlorinated pesticides and PCBs, method limits of detection (ng/g dry weight).<sup>Δ</sup>

Compound	1986 <sup>▼</sup>	1987 <sup>◆</sup>	1988 <sup>■</sup>	1989 <sup>▲</sup>	1990 <sup>*</sup>	1991-1992 <sup>†</sup>
Aldrin	0.16	0.07	0.03	0.07	0.24	0.42
<i>cis</i> -Chlordane	0.08	0.10	0.09	0.06	0.10	0.39
Dieldrin	0.16	0.14	0.03	0.07	0.10	0.40
Heptachlor	0.14	0.24	0.04	0.04	0.35	0.54
Heptachlor epoxide	0.16	0.08	0.03	0.06	0.15	0.46
Hexachlorobenzene	0.14	0.09	0.09	0.03	0.29	0.28
gamma-HCH	0.14	0.03	0.05	0.02	0.12	0.20
Mirex	0.20	0.09	0.05	0.13	0.22	0.49
<i>trans</i> -Nonachlor	0.20	0.09	0.06	0.07	0.22	0.42
2,4'-DDD	0.20	0.36	0.38	0.04	0.17	0.49
4,4'-DDD	0.49	0.10	0.17	0.05	0.25	0.58
2,4'-DDE	0.14	0.05	0.06	0.07	0.08	0.32
4,4'-DDE	0.16	0.07	0.07	0.06	0.13	0.25
2,4'-DDT	0.23	0.13	0.13	0.05	0.21	0.37
4,4'-DDT	0.08	0.27	0.03	0.05	0.24	0.62
Dichlorobiphenyls	0.62					
Trichlorobiphenyls	0.16					
Tetrachlorobiphenyls	0.16					
Pentachlorobiphenyls	0.14					
Hexachlorobiphenyls	0.42					
Heptachlorobiphenyls	0.14					
Octachlorobiphenyls	0.14					
Nonachlorobiphenyls	0.31					

<sup>Δ</sup> For 1990 and afterwards, detections limits are applicable to the East and the entire West Coasts.

<sup>▼</sup> Boehm *et al.* (1987); <sup>◆</sup> Boehm *et al.* (1988); <sup>■</sup> Freitas *et al.* (1989); <sup>▲</sup> Battelle (1990b); <sup>\*</sup> Battelle, 1991; <sup>†</sup> Battelle 1992.

Table A.26 (cont). Mussel Watch East and Northwest Coasts sediment chlorinated pesticides and PCBs, method limits of detection (ng/g dry weight).<sup>Δ</sup>

Compound	1986 <sup>▼</sup>	1987 <sup>◆</sup>	1988 <sup>■</sup>	1989 <sup>▲</sup>	1990 <sup>◇</sup>	1991-1992 <sup>†</sup>
PCB 8	-	0.07	0.16	0.07	0.36	0.87
PCB 18	-	0.09	0.06	0.06	0.27	0.48
PCB 28	-	0.06	0.12	0.07	0.23	0.23
PCB 44	-	0.11	0.08	0.07	0.21	0.67
PCB 52	-	0.12	0.05	0.11	0.26	0.26
PCB 66	-	0.13	0.11	0.08	0.22	0.43
PCB 77*	-	-	-	-	-	0.60
PCB 101	-	0.09	0.08	0.10	0.20	0.49
PCB 105	-	0.05	0.31	0.10	0.17	0.60
PCB 118	-	0.10	0.10	0.06	0.22	0.45
PCB 126*	-	-	-	-	-	0.60
PCB 128	-	0.54	0.33	0.09	0.16	0.34
PCB 138	-	0.08	0.11	0.08	0.20	0.45
PCB 153	-	0.10	0.08	0.10	0.30	0.82
PCB 170	-	0.12	0.03	0.10	1.84	0.67
PCB 180	-	0.11	0.02	0.12	0.26	0.49
PCB 187	-	0.39	0.22	0.08	0.28	0.58
PCB 195	-	0.19	0.02	0.09	0.17	0.61
PCB 206	-	0.19	0.02	0.09	0.24	0.96
PCB 209	-	0.17	0.02	0.12	0.24	0.63

<sup>Δ</sup> For 1990 and afterwards, detections limits are applicable to the East and the entire West Coasts.

\* PCB 77 and 126 were first quantified in the NS&T MWP in 1990.

<sup>▼</sup> Boehm *et al.* (1987); <sup>◆</sup> Boehm *et al.* (1988); <sup>■</sup> Freitas *et al.* (1989); <sup>▲</sup> Battelle (1990b); <sup>◇</sup> Battelle, 1991; <sup>†</sup> Battelle 1992.

Table A.27. Mussel Watch Project East and Northwest Coasts tissue aromatic hydrocarbons, method limits of detections (ng/g dry weight).<sup>Δ</sup>

Compound	1986▼	1987◆	1988■ ◇	1988■	1989▲	1990*	1990*
	FID	Full Scan Mode	Full Scan Mode	SIM Mode	SIM Mode	SIM (mussel)	SIM (oyster)
Acenaphthene	7.5	43	43	5.9	1.6	8.5	22
Acenaphthylene	-	-	36	3.6	11	36	16
Anthracene	10	48	48	8.8	7.2	23	28
Benz[ <i>a</i> ]anthracene	15	23	23	5.0	9.4	8.0	10
Dibenz[ <i>a,h</i> ]anthracene	8.5	83	83	6.8	5.8	7.4	9.9
Benzo[ <i>b</i> ]fluoranthene	-	-	17	3.6	16	12	14
Benzo[ <i>k</i> ]fluoranthene	-	-	14	4.8	6.3	18	22
Benzo[ <i>ghi</i> ]perylene	-	-	30	6.6	6.2	3.3	21
Benzo[ <i>a</i> ]pyrene	5.0	28	28	5.6	3.8	8.7	18
Benzo[ <i>e</i> ]pyrene	5.0	14	14	3.5	4.7	7.6	20
Biphenyl	3.0	40	40	14	3.7	41	40
Chrysene	40	32	32	5.5	3.1	12	18
2,6-Dimethylnaphthalene	8.5	50	50	4.7	6.0	38	15
Fluoranthene	18.0	40	41	3.2	8.2	25	34
Fluorene	3.0	27	27	6.2	2.9	12	23
Indeno[1,2,3- <i>cd</i> ]pyrene	-	-	65	8.0	8.5	5.5	22
1-Methylnaphthalene	21	53	53	7.6	3.5	22	15
2-Methylnaphthalene	8.5	48	48	7.5	4.0	20	16
1-Methylphenanthrene	3.0	30	30	4.3	7.1	22	28
Naphthalene	14	20	20	2.3	2.6	12	18
Perylene	6.0	22	22	6.3	2.2	11	20
Phenanthrene	8.5	37	37	5.7	5.2	24	30
Pyrene	16	100	100	4.7	8.0	20	37
1,6,7-Trimethylnaphthalene	-	58	58	3.7	4.4	9.7	21

<sup>Δ</sup> For 1990 and afterwards, detections limits are applicable to the East and the entire West Coasts.

▼ Boehm *et al.* (1987); ◆ Boehm *et al.* (1988); ■ Freitas *et al.* (1989); ▲ Battelle (1990b); \* Battelle, 1991.

◇ These are informational values. Beginning in 1988, all PAHs were quantified with GC/MS in the selected ion mode.

Table A.27 (cont). Mussel Watch Project East and Northwest Coasts tissue aromatic hydrocarbons, method limits of detections (ng/g dry weight).<sup>Δ</sup>

Compound	1991 <sup>†</sup>		1992 <sup>◇</sup>	
	SIM Mussel	SIM Oyster	SIM Mussel	SIM Oyster
Acenaphthene	6.1	14	3.8	6.1
Acenaphthylene	11	16	10	3.5
Anthracene	5.0	13	5.5	7.9
Benz[ <i>a</i> ]anthracene	8.4	26	8.6	14
Dibenz[ <i>a,h</i> ]anthracene	6.1	17	7.2	8.0
Benzo[ <i>b</i> ]fluoranthene	8.5	47	9.8	11
Benzo[ <i>k</i> ]fluoranthene	7.1	32	4.6	10
Benzo[ <i>ghi</i> ]perylene	12	22	6.7	8.3
Benzo[ <i>a</i> ]pyrene	7.4	25	3.6	5.6
Benzo[ <i>e</i> ]pyrene	10	24	4.3	8.0
Biphenyl	4.1	18	8.8	3.9
Chrysene	5.9	26	9.9	18
2,6-Dimethylnaphthalene	6.6	16	8.6	4.4
Fluoranthene	4.9	30	9.5	14
Fluorene	6.6	13	6.4	5.5
Indeno[1,2,3- <i>cd</i> ]pyrene	8.9	12	7.9	12
1-Methylnaphthalene	5.6	14	5.7	4.1
2-Methylnaphthalene	5.4	14	5.5	6.2
1-Methylphenanthrene	7.5	24	9.3	6.6
Naphthalene	11.5	11	11	3.7
Perylene	5.5	30	5.1	6.8
Phenanthrene	5.1	18	7.1	5.0
Pyrene	6.0	28	6.8	10
1,6,7-Trimethylnaphthalene	5.5	14	5.0	3.5

<sup>Δ</sup> For 1990 and afterwards, detections limits are applicable to the East and the entire West Coasts.

<sup>†</sup> Battelle, 1992; <sup>◇</sup> Battelle, in press.

Table A.28. Mussel Watch Project East and Northwest Coasts tissue pesticides and PCBs, method limits of detection (ng/g dry weight).<sup>Δ</sup>

Compound	1986▼	1987◆	1988■	1989▲	1990*	1990*
Aldrin	0.60	0.25	0.12	0.38	0.39	0.78
<i>cis</i> -Chlordane	0.30	0.36	0.32	0.32	1.3	1.7
Dieldrin	0.60	0.52	0.11	0.39	2.9	2.4
Heptachlor	0.50	0.91	0.15	0.21	1.3	1.1
Heptachlor epoxide	0.60	0.29	0.10	0.34	1.4	1.1
Hexachlorobenzene	0.50	0.32	0.34	0.18	0.74	1.4
gamma-HCH	0.50	0.10	0.18	0.11	1.9	0.74
Mirex	0.75	0.34	0.19	0.76	0.86	1.2
<i>trans</i> -Nonachlor	0.75	0.34	0.22	0.39	0.99	0.73
2,4'-DDD	0.75	1.3	1.4	0.21	0.69	0.50
4,4'-DDD	1.8	0.38	0.64	0.27	7.1	1.1
2,4'-DDE	0.50	0.17	0.24	0.39	1.1	1.8
4,4'-DDE	0.60	0.28	0.27	0.34	6.4	2.5
2,4'-DDT	0.85	0.50	0.49	0.27	1.1	0.79
4,4'-DDT	0.30	1.0	0.11	0.29	1.7	3.7
Dichlorobiphenyls	2.3					
Trichlorobiphenyls	0.60					
Tetrachlorobiphenyls	0.60					
Pentachlorobiphenyls	0.50					
Hexachlorobiphenyls	1.6					
Heptachlorobiphenyls	0.50					
Octachlorobiphenyls	0.50					
Nonachlorobiphenyls	1.2					
PCB 8		0.27	0.60	0.41	0.99	5.8
PCB 18		0.35	0.22	0.34	1.1	1.3
PCB 28		0.22	0.42	0.38	0.92	0.67
PCB 44		0.41	0.25	0.42	2.0	1.5
PCB 52		0.43	0.17	0.60	1.4	0.97
PCB 66		0.50	0.43	0.46	1.7	1.5
PCB 101		0.35	0.28	0.55	2.6	1.2
PCB 105		0.17	1.2	0.54	2.0	1.2
PCB 118		0.36	0.38	0.34	2.4	1.2
PCB 128		2.0	1.2	0.48	1.0	0.65
PCB 138		0.30	0.43	0.45	3.7	2.0
PCB 153		0.37	0.29	0.58	5.8	2.4
PCB 170		0.47	0.11	0.57	0.66	0.49
PCB 180		0.42	0.08	0.70	1.6	0.96
PCB 187		1.5	0.81	0.51	1.1	1.7
PCB 195		0.71	0.09	0.53	0.44	0.56
PCB 206		0.72	0.08	0.49	0.88	0.86
PCB 209		0.63	0.06	0.69	0.88	1.2

<sup>Δ</sup> For 1990 and afterwards, detections limits are applicable to the East and the entire West Coasts.

▼ Boehm *et al.* (1987); ◆ Boehm *et al.* (1988); ■ Freitas *et al.* (1989); ▲ Battelle (1990b); \* Battelle, 1991.

Table A.28 (cont). Mussel Watch Project East and Northwest Coasts tissue pesticides and PCBs, method limits of detection (ng/g dry weight).<sup>Δ</sup>

Compound	1991 <sup>†</sup>	1991 <sup>†</sup>	1992 <sup>◇</sup>	1992 <sup>◇</sup>
	Mussel	Oyster	Mussel	Oyster
Aldrin	0.68	1.4	0.66	1.4
<i>cis</i> -Chlordane	0.36	1.4	1.2	2.1
Dieldrin	7.3	2.4	0.82	0.74
Heptachlor	1.4	3.2	1.1	2.3
Heptachlor epoxide	0.78	1.2	0.96	2.2
Hexachlorobenzene	0.90	2.4	0.56	1.9
gamma-HCH	0.70	1.9	1.0	1.3
Mirex	0.52	2.7	0.55	0.10
<i>trans</i> -Nonachlor	0.47	1.5	1.0	2.5
2,4'-DDD	0.37	2.2	0.58	1.0
4,4'-DDD	0.47	2.4	1.0	0.76
2,4'-DDE	0.74	0.79	1.1	1.8
4,4'-DDE	0.95	1.8	2.3	0.55
2,4'-DDT	0.34	1.8	0.90	0.84
4,4'-DDT	1.5	8.2	2.1	1.1
PCB 8	4.0	6.8	2.8	4.2
PCB 18	2.8	4.0	1.6	2.9
PCB 28	1.1	2.8	0.80	3.4
PCB 44	1.0	2.6	1.2	3.1
PCB 52	1.7	5.1	1.4	3.3
PCB 66	0.61	1.3	0.97	4.4
PCB 77	0.95	3.1	1.2	1.5
PCB 101	0.60	1.9	1.5	3.2
PCB 105	0.52	1.1	0.46	1.7
PCB 118	0.49	1.7	2.4	1.6
PCB 126	0.74	3.0	0.98	0.66
PCB 128	1.1	0.80	1.2	0.89
PCB 138	0.63	2.8	0.65	1.2
PCB 153	0.88	1.2	2.1	0.65
PCB 170	1.4	5.6	0.79	0.69
PCB 180	0.46	1.4	1.2	1.6
PCB 187	0.63	2.2	0.67	1.4
PCB 195	1.5	1.6	0.49	0.55
PCB 206	0.94	1.7	0.68	0.44
PCB 209	2.8	5.2	0.69	0.42

<sup>Δ</sup> For 1990 and afterwards, detections limits are applicable to the East and the entire West Coasts.

<sup>†</sup>Battelle, 1992; <sup>◇</sup> Battelle, in press.

Table A.29. Mussel Watch Project East and West Coasts tissue organotin, method limits of detection (ng/g cation, dry weight).<sup>Δ</sup>

Compound	1988 <sup>■</sup>	1989-1991 <sup>▲</sup>	1992 <sup>†</sup>	
			Mussels	Oysters
Monobutyltin (MBT)	0.69	1.3	12	14
Dibutyltin (DBT)	1.1	1.4	12	11
Tributyltin (TBT)	25	3.1	19	31
Tetrabutyltin	-	1.6	6.7	15

<sup>Δ</sup> All butyltin analyses for the U.S. East and West Coasts were performed by Battelle, after 1987.

<sup>■</sup> Freitas *et al.* (1989). This was the first year TBT and its metabolites was analyzed by Battelle. These are the lowest reported values. <sup>▲</sup> Battelle (1990b). <sup>†</sup>Battelle, in press.

Table A.30. Mussel Watch Project California and Hawaii Coasts sediment aromatic hydrocarbons, method limits of detection (ng/g dry weight).<sup>Δ</sup>

Compound	1986 <sup>▼</sup>	1987 <sup>◆</sup>	1988 <sup>■</sup>	1989 <sup>▲</sup>
Acenaphthene	0.45	0.45	4.2	14
Acenaphthylene	-	-	3.0	15
Anthracene	1.7	1.7	4.1	15
Benz[ <i>a</i> ]anthracene	1.5	1.5	4.4	4.1
Dibenz[ <i>a,h</i> ]anthracene	0.90	0.90	8.5	19
Benzo[ <i>b</i> ]fluoranthene	-	-	7.8	8.6
Benzo[ <i>k</i> ]fluoranthene	-	-	5.9	7.5
Benzo[ <i>ghi</i> ]perylene	-	-	18	14
Benzo[ <i>a</i> ]pyrene	0.90	0.90	3.8	11
Benzo[ <i>e</i> ]pyrene	1.4	1.4	10	8.5
Biphenyl	0.75	0.75	3.9	18
Chrysene	1.5	1.5	6.6	10
2,6-Dimethylnaphthalene	1.2	1.2	3.9	15
Fluoranthene	1.7	1.7	3.7	9.9
Fluorene	0.30	0.30	2.2	14
Indeno[1,2,3- <i>cd</i> ]pyrene	-	-	16	12
1-Methylnaphthalene	0.90	0.90	7.0	3.7
2-Methylnaphthalene	0.75	0.75	3.0	3.8
1-Methylphenanthrene	2.1	2.1	4.9	13
Naphthalene	0.90	0.90	4.0	0.8
Perylene	1.2	1.2	5.5	10
Phenanthrene	1.2	1.2	4.0	18
Pyrene	2.0	2.0	3.3	13
1,6,7-Trimethylnaphthalene	-	-	2.4	13

<sup>Δ</sup> After 1989, all East and West Coast analyses were performed by Battelle.

<sup>▼</sup> Boehm *et al.* (1987). <sup>◆</sup> Boehm *et al.* (1988). <sup>■</sup> Freitas *et al.* (1989). <sup>▲</sup> Battelle (1990b).

Table A.31. Mussel Watch Project California and Hawaii Coasts sediment pesticides and PCBs, method limits of detection (ng/g dry weight).<sup>Δ</sup>

Compound	1986 ▼	1987 ◆	1988 ■	1989 ▲
Aldrin	0.06	0.06	0.04	0.04
<i>cis</i> -Chlordane	0.04	0.04	0.10	0.10
Dieldrin	0.07	0.07	0.11	0.11
Heptachlor	0.04	0.04	0.03	0.03
Heptachlor epoxide	0.05	0.05	0.07	0.07
Hexachlorobenzene	0.04	0.04	0.02	0.01
gamma-HCH	0.08	0.08	0.03	0.03
Mirex	0.04	0.04	0.23	0.23
<i>trans</i> -Nonachlor	0.05	0.05	0.10	0.10
2,4'-DDD	0.26	0.26	0.16	0.15
4,4'-DDD	0.18	0.18	0.21	0.21
2,4'-DDE	0.12	0.12	0.12	0.12
4,4'-DDE	0.20	0.20	0.15	0.15
2,4'-DDT	0.15	0.15	0.17	0.17
4,4'-DDT	0.09	0.09	0.21	0.21
Dichlorobiphenyls	0.18	0.18		
Trichlorobiphenyls	0.05	0.05		
Tetrachlorobiphenyls	0.11	0.11		
Pentachlorobiphenyls	0.11	0.11		
Hexachlorobiphenyls	0.09	0.09		
Heptachlorobiphenyls	0.06	0.06		
Octachlorobiphenyls	0.10	0.10		
Nonachlorobiphenyls	0.28	0.28		
PCB 8			0.01	0.01
PCB 18			0.03	0.03
PCB 28			0.06	0.06
PCB 44			0.08	0.08
PCB 52			0.06	0.06
PCB 66			0.14	0.16
PCB 101			0.14	0.13
PCB 105			0.24	0.24
PCB 118			0.21	0.21
PCB 128			0.24	0.24
PCB 138			0.23	0.23
PCB 153			0.19	0.19
PCB 170			0.28	0.28
PCB 180			0.28	0.28
PCB 187			0.23	0.23
PCB 195			0.31	0.31
PCB 206			0.32	0.32
PCB 209			0.39	0.39

<sup>Δ</sup> After 1989, all East and West Coast analyses were performed by Battelle.

▼ Boehm *et al.* (1987). ◆ Boehm *et al.* (1988). ■ Freitas *et al.* (1989). ▲ Battelle (1990b).

Table A.32. Mussel Watch Project California and Hawaii Coasts tissue aromatic hydrocarbons, method limits of detection (ng/g dry weight).<sup>Δ</sup>

Compound	1986 ▼	1987 ◆	1988 ■	1989 ▲
Acenaphthene	7.0	7.0	16	51
Acenaphthylene	-	-	11	54
Anthracene	28	28	15	55
Benz[ <i>a</i> ]anthracene	23	23	16	15
Dibenz[ <i>a,h</i> ]anthracene	20	20	31	69
Benzo[ <i>b</i> ]fluoranthene	-	-	29	32
Benzo[ <i>k</i> ]fluoranthene	-	-	22	28
Benzo[ <i>ghi</i> ]perylene	-	-	66	50
Benzo[ <i>a</i> ]pyrene	17	17	14	39
Benzo[ <i>e</i> ]pyrene	9.0	9.0	38	31
Biphenyl	8.0	8.0	15	66
Chrysene	20	20	25	38
2,6-Dimethylnaphthalene	5.0	5.0	14	57
Fluoranthene	19	19	14	37
Fluorene	9.0	9.0	8.0	62
Indeno[1,2,3- <i>cd</i> ]pyrene	-	-	57	46
1-Methylnaphthalene	1.0	1.0	26	14
2-Methylnaphthalene	1.0	1.0	11	14
1-Methylphenanthrene	23	23	18	47
Naphthalene	4.0	4.0	15	2.9
Perylene	20	20	20	37
Phenanthrene	14	14	15	48
Pyrene	47	47	12	48
1,6,7-Trimethylnaphthalene	-	-	8.8	47

<sup>Δ</sup> After 1989, all East and West Coast analyses were performed by Battelle.

▼ Boehm *et al.* (1987). ◆ Boehm *et al.* (1988). ■ Freitas *et al.* (1989). ▲ Battelle (1990b).

Table A.33. Mussel Watch Project California and Hawaii Coasts tissue pesticides and PCBs, method limits of detection (ng/g dry weight).<sup>Δ</sup>

Compound	1986 ▼	1987 ◆	1988 ■	1989 ▲
Aldrin	1.3	1.3	0.16	0.16
<i>cis</i> -Chlordane	0.06	0.06	0.35	0.35
Dieldrin	0.16	0.16	0.42	0.42
Heptachlor	0.09	0.09	0.10	0.10
Heptachlor epoxide	0.06	0.06	0.25	0.25
Hexachlorobenzene	0.72	0.72	0.06	0.05
gamma-HCH	0.03	0.03	0.11	0.11
Mirex	0.26	0.26	0.85	0.85
<i>trans</i> -Nonachlor	0.08	0.08	0.37	0.37
2,4'-DDD	0.21	0.21	0.57	0.57
4,4'-DDD	0.31	0.31	0.79	0.79
2,4'-DDE	0.10	0.10	0.44	0.44
4,4'-DDE	0.10	0.10	0.54	0.54
2,4'-DDT	0.64	0.64	0.62	0.62
4,4'-DDT	0.20	0.20	0.79	0.79
Dichlorobiphenyls	0.32	0.32		
Trichlorobiphenyls	1.2	1.2		
Tetrachlorobiphenyls	0.16	0.16		
Pentachlorobiphenyls	0.16	0.16		
Hexachlorobiphenyls	0.16	0.16		
Heptachlorobiphenyls	0.10	0.10		
Octachlorobiphenyls	1.6	1.6		
Nonachlorobiphenyls	0.16	0.16		
PCB 8			0.05	0.05
PCB 18			0.09	0.09
PCB 28			0.24	0.24
PCB 44			0.29	0.29
PCB 52			0.22	0.22
PCB 66			0.50	0.60
PCB 101			0.50	0.50
PCB 105			0.87	0.87
PCB 118			0.77	0.77
PCB 128			0.88	0.88
PCB 138			0.84	0.84
PCB 153			0.71	0.71
PCB 170			1.0	1.0
PCB 180			1.0	1.0
PCB 187			0.84	0.84
PCB 195			1.2	1.2
PCB 206			1.2	1.2
PCB 209			1.5	1.5

<sup>Δ</sup> After 1989, all East and West Coast analyses were performed by Battelle.

▼ Boehm *et al.* (1987). ◆ Boehm *et al.* (1988). ■ Freitas *et al.* (1989). ▲ Battelle (1990b).

Table A.34. Mussel Watch Project Gulf Coast sediment aromatic hydrocarbons, method limits of detection (ng/g dry weight).

Compound	1986 ▼	1987 ◆	1988 ■	1989-1992 ▲
Acenaphthene	5	5	5	4.5
Acenaphthylene	-	5	5	3.7
Anthracene	5	5	5	4.1
Benz[ <i>a</i> ]anthracene	5	5	5	1.4
Benzo[ <i>b</i> ]fluoranthene	-	5 <sup>Δ</sup>	5	1.8
Benzo[ <i>k</i> ]fluoranthene	-	-	5	1.9
Benzo[ <i>ghi</i> ]perylene	-	5	5	0.3
Benzo[ <i>a</i> ]pyrene	5	5	5	1.2
Benzo[ <i>e</i> ]pyrene	5	5	5	2.4
Biphenyl	5	5	5	2.0
Chrysene	5	5	5	0.5
Dibenz[ <i>a,h</i> ]anthracene	5	5	5	2.6
2,6-Dimethylnaphthalene	5	5	5	2.4
Fluoranthene	5	5	5	0.4
Fluorene	5	5	5	2.5
Indeno[1,2,3- <i>cd</i> ]pyrene	-	5	5	1.6
1-Methylnaphthalene	5	5	5	0.8
2-Methylnaphthalene	5	5	5	0.8
1-Methylphenanthrene	5	5	5	0.6
Naphthalene	5	5	5	0.5
Perylene	5	5	5	3.3
Phenanthrene	5	5	5	0.5
Pyrene	5	5	5	3.1
1,6,7-Trimethylnaphthalene	-	5	5	2.4

▼ Brooks *et al.* (1987). ◆ Brooks *et al.* (1988) and additional analyte data derived from the NS&T database. ■ Brooks *et al.* (1989). ▲ Brooks *et al.* (1990).

<sup>Δ</sup>In 1987, benzo[*b*]fluoranthene and benzo[*k*]fluoranthene were reported together as benzofluoranthene.

Table A.35. Mussel Watch Project Gulf Coast sediment pesticides and PCBs, method limits of detection (ng/g dry weight).

Compound	86▼	87-88◆■	89-92	Compound	86▼	87-88◆■	89-92
Aldrin	0.02	0.02	0.25	PCB 8	*	0.02	0.08
<i>cis</i> -Chlordane	0.02	0.02	0.23	PCB 18		0.02	0.25
Dieldrin	0.02	0.02	0.16	PCB 28		0.02	0.09
Heptachlor	0.02	0.02	0.20	PCB 44		0.02	0.09
Heptachlor epoxide	0.02	0.02	0.16	PCB 52		0.02	0.09
Hexachlorobenzene	0.02	0.02	0.37	PCB 66		0.02	0.14
gamma-HCH	0.02	0.02	0.22	PCB 101		0.02	0.13
Mirex	0.02	0.02	0.17	PCB 105		0.02	0.10
<i>trans</i> -Nonachlor	0.02	0.02	0.10	PCB 118		0.02	0.12
2,4'-DDD	0.02	0.02	0.13	PCB 128		0.02	0.13
4,4'-DDD	0.02	0.02	-	PCB 138		0.02	0.18
2,4'-DDE	0.02	0.02	0.28	PCB 153		0.02	0.12
4,4'-DDE	0.02	0.02	0.85	PCB 170		0.02	0.81
2,4'-DDT	0.02	0.02	0.25	PCB 180		0.02	0.16
4,4'-DDT	0.02	0.02	0.24	PCB 187		0.02	0.14
				PCB 195		0.02	0.25
				PCB 206		0.02	0.09
				PCB 209		0.02	0.78

▼ Brooks *et al.* (1987). ◆ Brooks *et al.* (1988) and additional analyte data derived NS&T database. ■ Brooks *et al.* (1989). ▲ Brooks *et al.* (1990).

\*Detection limits for congeners used to calculate chlorination levels were 0.02 ng/g dry weight, in 1986.

Table A.36. Mussel Watch Project Gulf Coast tissue aromatic hydrocarbons, method limits of detection (ng/g dry weight).

Compound	1986 ▼	1987 ♦	1988 ■	1989-1990 ▲●	1991-1992†
Acenaphthene	20	20	20	10	0.66
Acenaphthylene	-	20	20	21	0.55
Anthracene	20	20	20	9.1	0.92
Benz[ <i>a</i> ]anthracene	20	20	20	25	0.37
Benzo[ <i>b</i> ]fluoranthene	-	20*	20	20	0.59
Benzo[ <i>k</i> ]fluoranthene	-	-	20	19	0.69
Benzo[ <i>ghi</i> ]perylene	-	20	20	15	0.61
Benzo[ <i>a</i> ]pyrene	20	20	20	22	0.70
Benzo[ <i>e</i> ]pyrene	20	20	20	19	0.57
Biphenyl	20	20	20	15	1.6
Chrysene	20	20	20	19	0.68
Dibenz[ <i>a,h</i> ]anthracene	20	20	20	20	0.39
2,6-Dimethylnaphthalene	20	20	20	26	0.46
Fluoranthene	20	20	20	6.3	0.53
Fluorene	20	20	20	13	0.87
Indeno[1,2,3- <i>cd</i> ]pyrene	-	20	20	23	0.61
1-Methylnaphthalene	20	20	20	25	0.71
2-Methylnaphthalene	20	20	20	36	0.63
1-Methylphenanthrene	20	20	20	29	0.64
Naphthalene	20	20	20	23	1.4
Perylene	20	20	20	9.9	3.2
Phenanthrene	20	20	20	11	0.61
Pyrene	20	20	20	9.0	0.66
1,6,7-Trimethylnaphthalene	-	20	20	22	0.54

▼ Brooks *et al.* (1987). ♦ Brooks *et al.* (1988) and additional analyte data derived NS&T database. ■ Brooks *et al.* (1989). ▲ Brooks *et al.* (1990). ● GERG (1992a). † GERG (1992b).

\* Benzo[*b*]fluoranthene and benzo[*k*]fluoranthene coeluted and concentrations were reported as a sum of both isomers.

Table A.37. Mussel Watch Project Gulf Coast tissue pesticides and PCBs, method limits of detection (ng/g dry weight).

Compound	1986-1988▼◆■	1989 ▲	1990 ●	1991-1992†
Aldrin	0.25	2.4	2.4	0.49
<i>cis</i> -Chlordane	0.25	2.5	2.5	0.75
Dieldrin	0.25	2.9	2.9	0.66
Heptachlor	0.25	2.1	2.1	0.52
Heptachlor epoxide	0.25	0.85	0.85	0.57
Hexachlorobenzene	0.25	0.60	0.60	0.54
gamma-HCH	0.25	2.6	2.6	0.33
Mirex	0.25	1.2	1.2	0.54
<i>trans</i> -Nonachlor	0.25	1.7	1.7	1.9
2,4'-DDD	0.25	1.9	1.9	0.64
4,4'-DDD	0.25	7.0	7.0	0.38
2,4'-DDE	0.25	3.7	3.7	0.30
4,4'-DDE	0.25	5.5	5.5	0.76
2,4'-DDT	0.25	2.7	2.7	0.47
4,4'-DDT	0.25	2.6	2.6	0.38
PCB 8	0.25	2.1	2.1	0.84
PCB 18	0.25	0.86	0.86	0.52
PCB 28	0.25	1.5	1.5	0.35
PCB 44	0.25	2.8	2.8	0.24
PCB 52	0.25	2.4	2.4	0.92
PCB 66	0.25	2.2	2.2	0.39
PCB 77/110	-	-	4.7	1.1
PCB 101	0.25	6.6	6.6	0.51
PCB 105	0.25	0.88	0.88	1.1
PCB 118	0.25	4.0	4.0	0.47
PCB 126	-	-	2.3	0.72
PCB 128	0.25	2.1	2.1	0.40
PCB 138	0.25	7.3	7.3	5.9
PCB 153	0.25	4.7	4.7	1.6
PCB 170	0.25	*	*	*
PCB 180	0.25	1.8	1.8	0.36
PCB 187	0.25	4.7	4.7	0.71
PCB 195	0.25	1.8	1.8	0.89
PCB 206	0.25	1.5	1.5	0.59
PCB 209	0.25	1.6	1.6	0.59

▼ Brooks *et al.* (1987). ◆ Brooks *et al.* (1988). ■ Brooks *et al.* (1989). ▲ Brooks *et al.* (1990). ● GERG (1992a). †GERG (1992b).

\* Not reported due to interference, of phthalates.

Table A.38. Mussel Watch Project Gulf Coast tissue organotin, method limits of detection (ng/g Sn, dry weight).

Compound	1987		1988 - 1992
	bivalves*	sediments <sup>Δ</sup>	bivalves <sup>◇</sup>
Monobutyltin (MBT)	10	8	4.8
Dibutyltin (DBT)	10	5	25
Tributyltin (TBT)	20	5	23
Tetrabutyltin-	-	-	7.2

\* Wade *et al.* (1988) lowest reported values. East and West Coast sites were also analyzed.

<sup>Δ</sup> Wade *et al.* (1990) lowest reported values. East and West Coast sites were also analyzed.

<sup>◇</sup>Detection limit data taken from the NS&T database. Butyltins were only quantified in bivalves after 1988.

Table A.39. Mussel Watch Project East and Northwest Coasts sediment major and trace elements, method limits of detection ( $\mu\text{g/g}$  dry weight).<sup>Δ</sup>

Elements	1986-1987 <sup>▼◆</sup>	1988 <sup>■</sup>	1989 <sup>▲</sup>	1990 <sup>●</sup>	1991-1992 <sup>†</sup>
Al	5000	1500	1500	8000	13000
Si	4000	-	-	18000	22000
Cr	6.0	6.0	6.0	20	16
Mn	6.0	-	-	73	220
Fe	6.0	3.0	3.0	1800	400
Ni	4.0	2.1	2.1	7.7	8.5
Cu	3.0	2.4	2.4	21	6.4
Zn	2.0	2.4	2.4	15	15
As	1.7	2.1	2.1	2.5	4.3
Se	0.13	1.0	0.06	0.24	1.3
Ag	0.02	0.01	0.01	0.03	0.05
Cd	0.08	0.02	0.01	0.01	0.12
Sn	1.0	0.03	0.06	2.2	0.37
Sb	0.8	-	-	0.17	0.3
Hg	0.01	0.008	0.009	0.01	0.04
Tl	0.2	-	-	-	-
Pb	3.0	2.2	2.2	5.3	2.2

<sup>Δ</sup> For 1990 and afterwards, detections limits are applicable to the East and the entire West Coasts.

<sup>▼</sup> Boehm *et al.* (1987). <sup>◆</sup> Boehm *et al.* (1988). <sup>■</sup> Freitas *et al.* (1989). <sup>▲</sup> Battelle (1990b). <sup>●</sup> Battelle (1991). <sup>†</sup>Battelle (1992).

Table A.40. Mussel Watch Project East and Northwest Coasts tissue major and trace elements, method limits of detection ( $\mu\text{g/g}$  dry weight).<sup>Δ</sup>

Elements	1986-1987 <sup>▼◆</sup>	1988 <sup>■</sup>	1989 <sup>▲</sup>	1990 <sup>●</sup>	†1991	◇1992
Al	27	60	0.78	160	47	34
Si	600	-	-	110	5000	400
Cr	0.21	0.12	0.10	0.63	0.37	0.4
Mn	3.0	-	-	2.8	0.28	1.8
Fe	30	5.0	5.0	180	33	26
Ni	0.23	0.25	0.07	1.5	0.96	0.54
Cu	3.0	3.0	3.0	11	3.7	2.7
Zn	2.0	5.0	5.0	1.0	37	27
As	1.5	2.0	2.0	5.0	1.7	1.2
Se	0.58	1.0	1.0	0.5	0.77	0.61
Ag	0.12	0.01	0.03	0.01	0.09	0.3
Cd	0.9	0.19	0.02	0.63	0.21	0.44
Sn	0.5	0.04	0.005	0.01	0.55	0.74
Sb	0.4	-	-	0.01	0.01	0.03
Hg	0.001	0.01	0.01	0.01	0.005	0.006
Tl	0.05	-	-	-	-	-
Pb	0.06	0.17	0.02	0.13	0.04	0.09

<sup>Δ</sup> For 1990 and afterwards, detections limits are applicable to the East and the entire West Coasts.

<sup>▼</sup>Boehm *et al.* (1987). <sup>◆</sup>Boehm *et al.* (1988). <sup>■</sup>Freitas *et al.* (1989). <sup>▲</sup>Battelle (1990b). <sup>●</sup>Battelle (1991). <sup>†</sup>Battelle (1992). <sup>◇</sup>Battelle (in press).

Table A.41. Mussel Watch Project California and Hawaii Coasts sediment major and trace elements, method limits of detection ( $\mu\text{g/g}$  dry weight).<sup>Δ</sup>

Elements	1986 ▼	1987 ◆	1988 ■	1989 ▲
Al	560	390	210	3.5
Si	600	18	-	-
Cr	1.2	0.77	2.8	0.35
Mn	5.0	12	-	-
Fe	16	45	24	52
Ni	0.42	0.46	1.9	0.65
Cu	0.77	0.77	0.22	0.09
Zn	2.3	2	5.0	3.3
As	2.9	0.24	0.52	0.78
Se	6.9	0.6	2.0	*
Ag	0.01	0.02	0.03	0.07
Cd	0.08	0.009	0.03	0.09
Sn	5.0	1.7	◇	◇
Sb	6.2	0.07	-	-
Hg	0.008	0.004	0.004	0.006
Tl	16	0.43	-	-
Pb	0.28	0.26	0.61	0.61

<sup>Δ</sup> All East and West Coast major and trace elements are quantified by one laboratory (Battelle) beginning in 1990.

▼ Boehm *et al.* (1987). ◆ Boehm *et al.* (1988). ■ Freitas *et al.* (1989). ▲ Battelle (1990b). ● Battelle (1991).

◇ All East and West Coast Sn analyses were performed by Battelle starting in 1988.

\* All East and West Coast Se analyses were performed by Battelle starting in 1989.

Table A.42. Mussel Watch Project California and Hawaii Coasts tissue major and trace elements, method limits of detection ( $\mu\text{g/g}$  dry weight).<sup>Δ</sup>

Elements	1986 ▼	1987 ◆	1988 ■	1989 ▲
Al	0.28/22*	2.9	1.4	0.5
Si	100	180	-	-
Cr	0.15	0.01	0.05	0.07
Mn	1.1	0.18	-	-
Fe	2.9	7.1	15	12
Ni	0.24	0.2	0.19	0.32
Cu	0.13/1.3*	0.12	0.14	0.04
Zn	1.0	1.	1.9	5.4
As	1.7	0.46	0.62	0.60
Se	0.51	0.18	0.36	†
Ag	0.01	0.03	0.01	0.03
Cd	0.01/0.70*	0.03	0.02	0.03
Sn	0.6	1	◇	◇
Sb	0.30	0.15	-	-
Hg	0.003	0.013	0.011	0.015
Tl	0.06	0.16	-	-
Pb	0.06	0.06	0.2	0.05

<sup>Δ</sup> All East and West Coast major and trace elements are quantified by one laboratory (Battelle) beginning in 1990.

▼ Boehm *et al.* (1987). ◆ Boehm *et al.* (1988). ■ Freitas *et al.* (1989). ▲ Battelle (1990b). ● Battelle (1991).

\*Detection limits for GFAA and FAA, respectively.

◇ All East and West Coast Sn analyses were performed by Battelle starting in 1988.

† All East and West Coast Se analyses were performed by Battelle starting in 1989.

Table A.43. Mussel Watch Project Gulf Coast sediment major and trace elements, method limits of detection ( $\mu\text{g/g}$  dry weight).

Elements	1986-1988*	1989 <sup>▲</sup>	1990 <sup>●</sup>	1991 <sup>◇</sup>	1992 <sup>†</sup>
Al	1500	103	103	-	444
Si	10000	-	-	-	-
Cr	5.0	1.0	1.0	-	0.11
Mn	1	6	6	-	-
Fe	500	149	149	-	40.5
Ni	1.0	0.07	0.07	-	0.72
Cu	5.0	0.04	0.04	-	0.44
Zn	2.0	1	1	-	2.2
As	1.5	0.09	0.09	-	0.29
Se	0.1	0.07	0.07	-	0.17
Ag	0.01	0.02	0.02	-	0.03
Cd	0.05	0.002	0.002	-	0.008
Sn	0.1	0.2	0.2	-	0.11
Sb	0.2	-	-	-	-
Hg	0.01	0.01	0.01	-	0.008
Pb	1.0	0.2	0.2	-	0.35

\*For 1986-8, the values are derived from lowest reported concentrations in the NS&T database.

<sup>◇</sup>No sediments were analyzed in 1991.

<sup>▲</sup> Brooks *et al.* (1990). <sup>●</sup> GERG (1992a). <sup>†</sup>GERG (1992c).

Table A.44. Mussel Watch Project Gulf Coast tissue inorganic method limits of detection ( $\mu\text{g/g}$  dry weight).\*

Elements	1986-1988*	1989 <sup>▲</sup>	1990 <sup>●</sup>	1991 <sup>†</sup>	1992 <sup>◇</sup>
Al	10	-	-	-	-
Si	100	-	-	-	-
Cr	0.1	0.04	0.04	0.03	0.12
Mn	5.0	2	2	1.0	-
Fe	50	8	8	3.0	13
Ni	0.5	0.08	0.08	-	0.17
Cu	5.0	4	4	0.07	0.25
Zn	50	1	1	0.44	1.9
As	2.0	0.1	0.1	0.17	0.17
Se	1.0	0.3	0.3	0.12	0.49
Ag	0.01	0.02	0.02	0.002	0.04
Cd	0.2	0.004	0.004	0.002	0.008
Sn	0.05	0.2	0.2	0.02	0.19
Sb	0.2	-	-	-	-
Hg	0.01	0.01	0.01	0.005	0.03
Pb	0.1	0.09	0.09	0.05	0.12

\*For 1986-8, the values are derived from lowest reported concentrations in the NS&T database.

<sup>▲</sup> Brooks *et al.* (1990). <sup>●</sup> GERG (1992a). <sup>†</sup>GERG (1992b). <sup>◇</sup>GERG (1992b).

Table A.45. National Benthic Surveillance Project sites (Lauenstein *et al.*, 1993).

Site code	Site name	State	Latitude (N)	Longitude (W)	Species* code
MACCI	Machias Bay, Chance Island	ME	44° 38.0'	67° 20.0'	LS
MACHI	Machias Bay, Hog Island	ME	44° 40.6'	67° 20.7'	LS
FRNLP	Frenchmans Bay, Long Porcupine Island	ME	44° 25.0'	68° 10.0'	LS
PNBCH	Penobscot Bay, Colt Head Island	ME	44° 15.0'	68° 50.0'	LS
PNBJI	Penobscot Bay, Job Island	ME	44° 12.8'	69° 00.7'	LS
PNBII	Penobscot Bay, Islesboro Island	ME	44° 19.6'	68° 51.7'	WF
JONPN	Johns Bay, Pemaquid Neck	ME	43° 50.5'	69° 31.2'	WF
CASGC	Casco Bay, Great Chebeague Island	ME	43° 45.0'	70° 05.0'	LS
CASCI	Casco Bay, Cousins Island	ME	43° 41.4'	70° 08.0'	LS,WF
CAPRI	Cape Elizabeth, Richmond Island	ME	43° 31.9'	70° 16.6'	WF
MERPI	Merrimac River, Plum Island	MA	42° 45.0'	70° 45.0'	WF
SALFP	Salem Harbor, Folger Point	MA	42° 32.2'	70° 49.6'	WF
BOSPR	Boston Harbor, President Roads	MA	42° 20.0'	70° 59.0'	WF
BOSDI	Boston Harbor, Deer Island	MA	42° 19.9'	70° 58.1'	WF
BOSQB	Boston Harbor, Quincy Bay	MA	42° 18.4'	70° 58.4'	WF
BOSHB	Boston Harbor, Hull Bay	MA	42° 17.1'	70° 54.4'	WF
BOSMR	Boston Harbor, Mystic River	MA	42° 23.2'	71° 03.2'	WF
BUZWI	Buzzards Bay, West Island	MA	41° 35.0'	70° 45.0'	WF
NBHCP	New Bedford Harbor, Clarks Point	MA	41° 35.0'	70° 53.5'	WF
NARCI	Narragansett Bay, Conanicut Island	RI	41° 35.0'	71° 22.0'	WF
NARPI	Narragansett Bay, Prudence Island	RI	41° 40.4'	71° 21.2'	WF
LISLS	Long Island Sound, Long Sand Shoal	NY	41° 12.0'	72° 20.0'	WF
LISRP	Long Island Sound, Rocky Point	NY	41° 08.7'	72° 24.7'	WF
LISON	Long Island Sound, Oak Neck Point	NY	40° 58.0'	73° 35.0'	WF
LISLP	Long Island Sound, Lloyd Point	NY	40° 58.5'	73° 28.9'	WF
RARLB	Raritan Bay, Lower Bay	NJ	40° 28.0'	74° 05.0'	WF
RARER	Raritan Bay, East Reach	NJ	40° 29.5'	74° 05.4'	WF
RARWR	Raritan Bay, West Reach	NY	40° 30.4'	74° 10.2'	WF
RARGB	Raritan Bay, Gravesend Bay	NY	40° 35.4'	74° 01.6'	WF
RARUB	Raritan Bay, Upper Bay	NY	40° 39.7'	74° 02.8'	WF
GRTSI	Great Bay, Seven Island	NJ	39° 31.0'	74° 23.0'	WF
GRTWI	Great Bay, Wells Island	NJ	39° 31.7'	74° 23.6'	WF
GRTIW	Great Bay Intracoastal Waterway	NJ	39° 26.7'	74° 23.5'	WF
DELBS	Delaware Bay, Brandywine Shoal	DE	39° 00.0'	75° 10.0'	WpF
DELTS	Delaware Bay, The Shears	DE	38° 52.8'	75° 10.4'	WpF
DELCI	Delaware Bay, Cherry Island Range	DE	39° 42.6'	75° 30.0'	WP
BALFM	Baltimore Harbor, Fort McHenry Channel	MD	39° 14.7'	76° 33.8'	SED
BALBC	Baltimore Harbor, Brewerton Channel	MD	39° 12.5'	76° 31.4'	WP
CHBCR	Chesapeake Bay, Chester River	MD	39° 01.6'	76° 11.9'	WP
CHBGI	Chesapeake Bay, Gibson Island	MD	39° 05.0'	76° 20.0'	SP
CHBKI	Chesapeake Bay, Kent Island	MD	39° 01.4'	76° 22.1'	SP
CHBSI	Chesapeake Bay, Smith Island	MD	37° 55.0'	76° 10.0'	SED
CHBYR	Chesapeake Bay, York River	VA	37° 10.0'	76° 10.0'	AC,SP
CHBER	Chesapeake Bay, Elizabeth River	VA	36° 50.8'	76° 18.0'	AC
PAMJB	Pamlico Sound, Jones Bay	NC	35° 13.5'	76° 32.1'	AC
CHSSC	Charleston Harbor, South Channel	SC	32° 45.4'	79° 54.4'	AC
CHSCO	Charleston Harbor, Coastal	SC	32° 50.1'	79° 40.2'	AC

Table A.45 (cont). National Benthic Surveillance Project sites (Lauenstein *et al.*, 1993).

Site code	Site name	State	Latitude (N)	Longitude (W)	Species* code
SAVEI	Savannah River, Elba Island	GA	32° 05.8'	80° 59.8'	AC, HC
SAPHP	Sapelo Sound, High Point	GA	31° 32.3'	81° 14.5'	SP
SAPBI	Sapelo Sound, Barbour Is. River	GA	31° 34.8'	81° 14.5'	AC
SAPDH	Sapelo Sound, Dog Hammock	GA	31° 31.9'	81° 17.5'	AC
SAPIN	Sapelo Sound, Inlet	GA	31° 32.5'	81° 11.8'	AC, SP
SAPSN	Sapelo Sound, South Newport River	GA	31° 38.6'	81° 15.4'	AC
SAPJC	Sapelo Sound, Johnson Creek	GA	31° 38.9'	81° 11.4'	AC
SJROP	St. Johns River, Orange Point	FL	30° 09.7'	81° 40.9'	AC
SJRTR	St. Johns River, Trout River	FL	30° 23.7'	81° 38.7'	SED
SJROR	St. Johns River, Ortega River	FL	30° 16.6'	81° 42.6'	AC
SJRMC	St. Johns River, W. Mill Cove	FL	30° 23.6'	81° 36.5'	SED
SJRPP	St. Johns River, Piney Point	FL	30° 14.4'	81° 39.4'	AC
SJRAC	St. Johns River, Arlington Channel	FL	30° 21.0'	81° 36.8'	SP, AC
SJRQI	St. Johns R., Quarantine Is. Upper Range	FL	30° 23.5'	81° 34.1'	SP, AC
BISNB	Biscayne Bay, North Bay	FL	25° 48.9'	80° 09.6'	PF
BISCK	Biscayne Bay, Chicken Key	FL	25° 36.9'	80° 17.6'	PF
LOTCH	Charlotte Harbor, Cape Haze	FL	26° 49.8'	82° 06.3'	SP
TAMTB	Tampa Bay, Northern Tampa Bay	FL	27° 46.8'	82° 34.0'	HC
APASG	Apalachicola Bay, St. George Island	FL	29° 38.9'	84° 58.4'	AC, SP
ANDMP	St. Andrews Bay, Military Point	FL	30° 07.6'	85° 38.0'	AC
COCCB	Choctawhatchee Bay, Choctawhatchee Bay	FL	30° 26.4'	86° 20.3'	AC
COCDH	Choctawhatchee Bay, Destin Harbor	FL	30° 23.4'	86° 29.8'	AC
PENPB	Pensacola Bay, Pensacola Bay	FL	30° 25.5'	87° 11.2'	AC, SP
MOBNP	Mobile Bay, North Point	AL	30° 17.8'	88° 04.8'	AC
MOBMR	Mobile Bay, Mobile River	AL	30° 38.2'	87° 59.2'	SED
PASPR	Pascagoula River, Pascagoula River	MS	30° 22.8'	88° 34.1'	AC
ROURI	Round Island, Round Island	MS	30° 18.4'	88° 36.6'	SP, AC
HERHB	Heron Bay, Heron Bay	MS	30° 11.0'	89° 28.5'	AC
MRDSP	Mississippi River, Delta, Southeast Pass	LA	29° 07.2'	89° 04.2'	AC
MRDHP	Mississippi River, Delta, Head of Passes	LA	29° 12.6'	89° 16.7'	AC, HC
BARBP	Barataria Bay, Barataria Pass	LA	29° 19.2'	89° 56.4'	AC
CALPL	Calcasieu River, Prien Lake	LA	30° 11.6'	93° 17.1'	AC, HC
CALWC	Calcasieu River, West Cove	LA	29° 52.4'	93° 22.2'	AC, HC
CALBI	Calcasieu River, Bayou d' Inde	LA	30° 12.6'	97° 18.1'	HC
GALEB	Galveston Bay, East Bay	TX	29° 27.3'	94° 42.8'	AC, HC, BD, RD
GALTC	Galveston Bay, Texas City	TX	29° 21.6'	94° 52.4'	AC
GALMP	Galveston Bay, Morgans Point	TX	29° 42.0'	94° 59.8'	AC, RD
GALEP	Galveston Bay, Eagle Point	TX	29° 29.9'	94° 53.7'	AC, SP, ST
GALTB	Galveston Bay, Trinity Bay	TX	29° 36.4'	94° 45.5'	AC
GALBB	Galveston Bay, Boggy Bayou	TX	29° 44.4'	95° 06.8'	AC
GALGB	Galveston Bay, Greens Bayou	TX	29° 44.6'	95° 09.8'	AC, SP, ST
GALGI	Galveston Bay, Goat Islands	TX	29° 44.9'	95° 03.8'	AC
GALCL	Galveston Bay, Clear Lake	TX	29° 33.3'	95° 02.7'	AC
LAVLB	Lavaca Bay, Lavaca Bay	TX	28° 38.8'	96° 36.0'	AC

Table A.45 (cont). National Benthic Surveillance Project sites (Lauenstein *et al.*, 1993).

Site code	Site name	State	Latitude (N)	Longitude (W)	Species* code
LAVPC	Lavaca Bay, Point Comfort	TX	28° 39.3'	96° 34.6'	BD, RD, ST, XT, HC
SABSB	San Antonio Bay, San Antonio Bay	TX	28° 13.2'	96° 46.4'	AC
CCBLR	Corpus Christi Bay, Long Reef	TX	27° 49.6'	97° 17.4'	AC, SP
CCBCC	Corpus Christi Bay, Corpus Christi Ch.	TX	27° 48.8'	97° 24.2'	SED
LLMLH	Lower Laguna Madre, Laguna Heights	TX	26° 06.5'	97° 15.4'	AC
SDBOU	San Diego Bay, Outside	CA	32° 38.0'	117° 11.0'	HT
SDBNC	San Diego Bay, National City	CA	32° 40.1'	117° 07.6'	BSB
SDBTE	San Diego Bay, Twenty Eighth Street	CA	32° 41.0'	117° 08.0'	BC, BSB, SSB
SDBNO	San Diego Bay, North	CA	32° 43.0'	117° 11.0'	WC
SDBHI	San Diego Bay, Harbor Island	CA	32° 43.4'	117° 12.7'	WC
SDBSI	San Diego Bay, Shelter Island	CA	32° 42.5'	117° 13.7'	BC
MIBOU	Mission Bay, Outside	CA	32° 47.1'	117° 15.5'	BC
OCEOU	Oceanside Harbor, Outside	CA	33° 11.6'	117° 23.7'	QF
DANOU	Dana Point Harbor, Outside	CA	33° 27.0'	117° 41.0'	BSB, HT, WC
DANIH	Dana Point, Inside Harbor	CA	33° 27.5'	117° 42.1'	BSB
SPBSB	San Pedro Bay, Seal Beach	CA	33° 44.0'	118° 08.0'	WC
SPBLB	San Pedro Bay, Long Beach	CA	33° 44.0'	118° 10.0'	WC
SPBOH	San Pedro Bay, Outer Harbor	CA	33° 42.6'	118° 15.4'	WC
SPBCC	San Pedro Bay, Cerritos Channel	CA	33° 45.7'	118° 15.3'	WC
SPBOU	San Pedro Bay, Outside	CA	33° 42.0'	118° 15.7'	WC, HT
SMBMB	Santa Monica Bay, Manhattan Beach	CA	33° 53.0'	118° 26.0'	HT
SMBWE	Santa Monica Bay, West	CA	33° 56.0'	118° 34.0'	HT
SMBSE	Santa Monica Bay, Southeast	CA	33° 47.5'	118° 27.0'	HT, WC
SMBSO	Santa Monica Bay, South	CA	33° 52.5'	118° 27.0'	HT
SMBNO	Santa Monica Bay, North	CA	33° 59.3'	118° 35.9'	HT
SMBDE	Santa Monica Bay, Deep	CA	33° 55.6'	118° 45.2'	SED
SLUOB	San Luis Obispo	CA	35° 06.1'	120° 45.9'	WC
ESTBY	Estero Bay	CA	35° 21.5'	121° 53.2'	SED
MONIH	Monterey Bay, Indian Head Beach	CA	36° 38.0'	121° 51.0'	ES
MONML	Monterey Bay, Moss Landing	CA	36° 48.0'	121° 48.0'	ES, SF
FARIS	Farallon Islands	CA	37° 39.4'	123° 03.5'	SED
SFBHP	San Francisco Bay, Hunters Point	CA	37° 42.0'	122° 22.0'	WC, SF
SFBRC	San Francisco Bay, Redwood City	CA	37° 33.4'	122° 11.2'	WC
SFBOA	San Francisco Bay, Oakland Entrance	CA	37° 47.5'	122° 20.3'	WC, PSS
SFBOE	San Francisco Bay, Oakland Estuary	CA	37° 47.0'	122° 21.0'	WC
SFBSS	San Francisco Bay, Southampton Shoal	CA	37° 53.0'	122° 24.0'	WC, SF
SFBCC	San Francisco Bay, Castro Creek	CA	37° 58.8'	122° 24.8'	SF
SFBSP	San Francisco Bay, San Pablo Bay	CA	38° 03.0'	122° 17.0'	SF
SFBIC	San Francisco Bay, Islais Creek Channel	CA	37° 44.9'	122° 22.1'	SED
BODNO	Bodega Bay, North	CA	38° 18.0'	123° 02.0'	WC, ES, SF
HUMII	Humboldt Bay, Indian Island	CA	40° 49.0'	124° 10.0'	SF
COONB	Coos Bay, North Bend	OR	43° 24.0'	124° 13.0'	SF, ES

Table A.45 (cont). National Benthic Surveillance Project sites (Lauenstein *et al.*, 1993).

Site code	Site name	State	Latitude (N)	Longitude (W)	Species*
COLYB	Columbia River, Youngs Bay	OR	46° 10.0'	123° 50.0'	SF
COLDS	Columbia River, Desdemona Sands	WA	46° 13.0'	123° 56.0'	SF
PUGNR	Puget Sound, Nisqually Reach	WA	47° 06.8'	122° 41.6'	ES
PUGCB	Puget Sound, Commencement Bay	WA	47° 17.0'	122° 25.3'	ES, FS
PUGEB	Puget Sound, Elliott Bay	WA	47° 36.0'	122° 21.0'	ES, FS
BOCBP	Boca de Quadra, Bacrian Point	AK	55° 16.0'	130° 33.0'	FS
LUTCR	Lutak Inlet, Chilkoot River Mouth	AK	59° 18.7'	135° 31.5'	FS
SKASR	Skagway, Skagway River	AK	59° 26.6'	135° 19.7'	FS
NAHES	Nahku Bay, East Side	AK	59° 28.0'	135° 20.0'	FS
PWSPV	Prince William Sound, Port Valdez	AK	61° 07.0'	146° 18.0'	FS
GOAKB	Gulf of Alaska, Kamishak Bay	AK	59° 15.0'	153° 42.0'	FS
BERDH	Bering Sea, Dutch Harbor	AK	53° 54.0'	166° 30.0'	FS
BERPM	Bering Sea, Port Moller	AK	56° 03.0'	160° 45.0'	FS
BERKB	Bering Sea, Kvichak Bay	AK	58° 41.0'	157° 36.0'	YfS
BERKR	Bering Sea, Kuskokwim River	AK	59° 54.0'	162° 15.0'	AF
BERYR	Bering Sea, Yukon River	AK	62° 55.0'	165° 23.0'	YfS, AF
NORNO	Norton Sound, Nome	AK	64° 19.2'	165° 30.3'	YfS
CHKRD	Chukchi Sea, Red Dog Mine	AK	67° 29.5'	164° 02.8'	SF
BEAOP	Beaufort Sea, Oliktok Point	AK	70° 30.0'	149° 58.0'	FhS
BEAPB	Beaufort Sea, Prudhoe Bay	AK	70° 21.0'	147° 57.0'	FhS

\* Species codes

AC - <i>Micropogonias undulatus</i> (Atlantic Croaker)	PSS - <i>Leptocottus armatus</i> (Pacific Staghorn Sculpin)
AF - <i>Liopsetta glacialis</i> (Arctic Flounder)	QF - <i>Seriphus politus</i> (Queenfish)
BC - <i>Cheilotrema saturnum</i> (Black Croaker)	RD - <i>Sciaenops ocellatus</i> (Red Drum)
BD - <i>Pogonias cromis</i> (Black Drum)	RS - <i>Lepidopsetta bilineata</i> (Rock Sole)
BSB - <i>Paralabrax nebulifer</i> (Barred Sand Bass)	SED - Sediment collection site only
CH - <i>Paralichthys californicus</i> (California Halibut)	SF - <i>Platichthys stellatus</i> (Starry Flounder)
CTf - <i>Symphurus atricauda</i> (California Tonguefish)	SP - <i>Leiostomus xanthurus</i> (Spot)
DT - <i>Hypsopsetta guttulata</i> (Diamond Turbot)	SpT - <i>Pleuroichthys ritteri</i> (Spotted Turbot)
ES - <i>Parophrys vetulus</i> (English Sole)	SSB - <i>Paralabrax maculatofasciatus</i> (Spotted Sandbass)
FhS - <i>Myoxocephalus quadricornis</i> (Four-horn Sculpin)	ST - <i>Cynoscion arenarius</i> (Sand Seatrout)
FS - <i>Hippoglossoides elassodon</i> (Flathead Sole)	WC - <i>Genyonemus lineatus</i> (White Croaker)
HC - <i>Arius felis</i> (Hardhead Catfish)	WF - <i>Pleuronectes americanus</i> (Winter Flounder)
HT - <i>Pleuronichthys verticalis</i> (Hornyhead Turbot)	WP - <i>Roccus americanus</i> (White Perch)
LS - <i>Myoxocephalus octodecemspinosus</i> (Longhorn Sculpin)	WpF - <i>Scophthalmus aquosus</i> (Windowpane Flounder)
LsC - <i>Coregonus sardinella</i> (Least Cisco)	WSP - <i>Phanerodon furcatus</i> (White Surf Perch)
PF - <i>Lagodon rhomboides</i> (Pinfish)	XT - <i>Cynoscion nebulosus</i> (Spotted Seatrout)
	YfS - <i>Limanda aspera</i> (Yellowfin Sole)

Table A.46. Mussel Watch Project sites (Lauenstein *et al.*, 1993).

Site Code	Site name	State	Latitude (N)	Longitude (W)	Species* code
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PBPI	Penobscot Bay, Pickering Island	ME	44° 15.88'	68° 44.05'	ME
PBSI	Penobscot Bay, Sears Island	ME	44° 27.13'	68° 53.38'	ME
MSSP	Merriconeag Sound, Stover Point	ME	43° 45.48'	69° 59.72'	ME
CAKP	Cape Arundel, Kennebunkport	ME	43° 20.87'	70° 28.48'	ME
CAGH	Cape Ann, Gap Head	MA	42° 39.65'	70° 35.71'	ME
			42° 40.04'	70° 36.30'	SED
SHFP	Salem Harbor, Folger Point	MA	42° 31.13'	70° 52.02'	ME
MBNB	Massachusetts Bay, Nahant Bay	MA	42° 25.23'	70° 54.41'	ME
			42° 25.58'	70° 54.10'	SED
BHDI	Boston Harbor, Deer Island	MA	42° 21.50'	70° 58.40'	ME
BHDB	Boston Harbor, Dorchester Bay	MA	42° 18.25'	71° 02.30'	ME
BHHB	Boston Harbor, Hingham Bay	MA	42° 16.45'	70° 53.26'	ME
BHBI	Boston Harbor, Brewster Island	MA	42° 20.55'	70° 52.68'	ME
MBNR	Massachusetts Bay, North River	MA	42° 09.65'	70° 44.41'	ME
DBCI	Duxbury Bay, Clarks Island	MA	42° 00.88'	70° 38.17'	ME
CCNH	Cape Cod, Nauset Harbor	MA	41° 47.68'	69° 56.90'	ME
BBNI	Buzzards Bay, Naushon Island	MA	41° 30.77'	70° 44.49'	ME
			41° 30.60'	70° 44.26'	SED
BBWF	Buzzards Bay, West Falmouth	MA	41° 36.50'	70° 39.35'	ME
			41° 36.77'	70° 40.37'	SED
BBCC	Buzzards Bay, Cape Cod Canal	MA	41° 44.37'	70° 37.02'	ME
BBAR	Buzzards Bay, Angelica Rock	MA	41° 34.63'	70° 51.78'	ME
			41° 35.22'	70° 52.70'	SED
BBRH	Buzzards Bay, Round Hill	MA	41° 32.45'	70° 55.52'	ME
BBGN	Buzzards Bay, Goosebury Neck	MA	41° 28.68'	71° 02.13'	ME
			41° 28.84'	71° 01.34'	SED
NBMH	Narragansett Bay, Mount Hope Bay	RI	41° 40.60'	71° 35.57'	SED
NBDI	Narragansett Bay, Dyer Island	RI	41° 36.20'	71° 17.37'	ME
NBPI	Narragansett Bay, Patience Island	RI	41° 39.37'	71° 21.13'	ME
NBDU	Narragansett Bay, Dutch Island	RI	41° 30.08'	71° 23.57'	ME
BIBI	Block Island Sound, Block Island	RI	41° 11.40'	71° 35.14'	ME
LICR	Long Island Sound, Connecticut River	CT	41° 15.83'	72° 20.50'	ME
LINH	Long Island Sound, New Haven	CT	41° 15.40'	72° 56.67'	ME, CV
LIHR	Long Island Sound, Housatonic River	CT	41° 10.07'	73° 06.58'	ME, CV
LISI	Long Island Sound, Sheffield Island	CT	41° 03.40'	73° 24.77'	ME
LIMR	Long Island Sound, Mamaroneck	NY	40° 56.47'	73° 42.03'	ME
LITN	Long Island Sound, Throgs Neck	NY	40° 49.17'	73° 48.07'	ME
LIHH	Long Island Sound, Hempstead Harbor	NY	40° 51.14'	73° 40.14'	ME
LIHU	Long Island Sound, Huntington Harbor	NY	40° 55.00'	73° 25.87'	ME
LIPJ	Long Island Sound, Port Jefferson	NY	40° 57.57'	73° 05.52'	ME, CV
LIGB	Long Island, Gardiners Bay	NY	40° 59.90'	72° 06.68'	ME
MBTH	Moriches Bay, Tuthill Point	NY	40° 46.65'	72° 45.37'	ME
LIFI	Long Island, Fire Island Inlet	NY	40° 37.68'	73° 17.16'	ME
LIJI	Long Island, Jones Inlet	NY	40° 35.81'	73° 35.45'	ME
HRJB	Hudson/Raritan Estuary, Jamaica Bay	NY	40° 34.13'	73° 53.88'	ME
HRUB	Hudson/Raritan Estuary, Upper Bay	NY	40° 41.38'	74° 02.55'	ME
HRLB	Hudson/Raritan Estuary, Lower Bay	NY	40° 33.97'	74° 03.13'	ME

Table A.46 (cont). Mussel Watch Project sites (Lauenstein *et al.*, 1993).

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Site Code	Site name	State	Latitude (N)	Longitude (W)	Species* code
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HRRB	Hudson/Raritan Estuary, Raritan Bay	NJ	40° 31.12'	74° 11.05'	ME
			40° 30.13'	74° 09.71'	SED
NYSH	New York Bight, Sandy Hook	NJ	40° 29.27'	74° 02.70'	ME
NYLB	New York Bight, Long Branch	NJ	40° 17.68'	73° 58.56'	ME
NYSR	New York Bight, Shark River	NJ	40° 11.18'	74° 00.38'	ME
BIBL	Barnegat Inlet, Barnegat Light	NJ	39° 45.52'	74° 05.93'	ME
AIAC	Absecon Inlet, Atlantic City	NJ	39° 22.15'	74° 24.48'	ME
DBCM	Delaware Bay, Cape May	NJ	38° 58.92'	74° 57.92'	ME
			38° 58.92'	74° 58.13'	SED
DBFE	Delaware Bay, False Egg Island Point	NJ	39° 12.82'	75° 11.45'	CV
			39° 12.71'	75° 11.45'	SED
DBBD	Delaware Bay, Ben Davis Pt. Shoal	NJ	39° 15.93'	75° 16.93'	CV
			39° 16.41'	75° 16.22'	SED
DBAP	Delaware Bay, Arnolds Point Shoal	NJ	39° 23.09'	75° 25.88'	CV
DBHC	Delaware Bay, Hope Creek	NJ	39° 25.60'	75° 29.60'	CV
DBWB	Delaware Bay, Woodland Beach	DE	39° 19.92'	75° 27.42'	CV
DBKI	Delaware Bay, Kelly Island	DE	39° 12.17'	75° 21.30'	CV
DBCH	Delaware Bay, Cape Henlopen	DE	38° 47.28'	75° 07.42'	ME
CBBO	Chesapeake Bay, Bodkin Point	MD	39° 09.60'	76° 24.07'	CV
CBMP	Chesapeake Bay, Mountain Point Bar	MD	39° 04.42'	76° 24.73'	CV
CBHP	Chesapeake Bay, Hackett Point Bar	MD	38° 58.37'	76° 25.00'	CV
CBCP	Chesapeake Bay, Choptank River	MD	38° 36.41'	76° 07.20'	CV
CBHG	Chesapeake Bay, Hog Point	MD	38° 18.74'	76° 23.87'	CV
PRSP	Potomac River, Swan Point	MD	38° 16.90'	76° 56.02'	CV
CBCI	Chincoteague Bay, Chincoteague Inlet	VA	37° 56.51'	75° 22.60'	CV
QIUB	Quinby Inlet, Upshur Bay	VA	37° 31.85'	75° 43.38'	CV
CBCC	Chesapeake Bay, Cape Charles	VA	37° 17.09'	76° 01.19'	CV
PRMC	Potomac River, Mattox Creek	VA	38° 13.12'	76° 57.32'	CV
PRRP	Potomac River, Ragged Point	VA	38° 09.37'	76° 35.87'	CV
CBIB	Chesapeake Bay, Ingram Bay	VA	37° 47.63'	76° 17.06'	CV
RRRR	Rappahannock River, Ross Rock	VA	37° 54.08'	76° 47.43'	CV
CBDP	Chesapeake Bay, Dandy Point	VA	37° 06.04'	76° 17.73'	CV
CBJR	Chesapeake Bay, James River	VA	37° 04.07'	76° 36.68'	CV
RSJC	Roanoke Sound, John Creek	NC	35° 53.47'	75° 37.98'	CV
PSWB	Pamlico Sound, Wysocking Bay	NC	35° 24.67'	76° 03.45'	CV
PSPR	Pamlico Sound, Pungo River	NC	35° 19.48'	76° 26.95'	CV
PSNR	Pamlico Sound, Neuse River	NC	35° 05.42'	76° 31.65'	CV
PSCH	Pamlico Sound, Cape Hatteras	NC	35° 12.68'	75° 43.24'	CV
			35° 12.37'	75° 42.96'	SED
BIPI	Beaufort Inlet, Pivers Island	NC	34° 43.10'	76° 40.53'	CV
CFBI	Cape Fear, Battery Island	NC	33° 54.92'	78° 00.50'	CV
WBLB	Winyah Bay, Lower Bay	SC	33° 14.60'	79° 11.78'	CV
SRNB	Santee River, North Bay	SC	33° 10.37'	79° 14.92'	CV
CHFJ	Charleston Harbor, Fort Johnson	SC	32° 45.32'	79° 52.70'	CV
CHSF	Charleston Harbor, Shutes Folly Island	SC	32° 46.83'	79° 55.00'	CV
SRTI	Savannah River Estuary, Tybee Island	GA	32° 01.20'	80° 52.25'	CV

Table A.46 (cont). Mussel Watch Project sites (Lauenstein *et al.*, 1993).

Site Code	Site name	State	Latitude (N)	Longitude (W)	Species* code
SSSI	Sapelo Sound, Sapelo Island	GA	31° 23.20'	81° 17.33'	CV
ARWI	Altamaha River, Wolfe Island	GA	31° 19.37'	81° 18.48'	CV
			31° 19.62'	81° 19.50'	SED
SJCB	St. Johns River, Chicopit Bay	FL	30° 22.62'	81° 26.63'	CV
MRCB	Matanzas River, Crescent Beach	FL	29° 46.00'	81° 15.38'	CV
IRSR	Indian River, Sebastian River	FL	27° 50.09'	80° 28.65'	CV
			27° 51.06'	80° 28.70'	SED
NMML	North Miami, Maule Lake	FL	25° 56.13'	80° 08.77'	CV
BBGC	Biscayne Bay, Gould's Canal	FL	25° 31.39'	80° 18.85'	CV
BBPC	Biscayne Bay, Princeton Canal	FL	25° 31.13'	80° 19.75'	CV
BHKF	Bahia Honda, Florida Keys	FL	24° 39.52'	81° 16.43'	CS
PRBB	Bahia de Boqueron, Puerto Rico	PR	18° 00.44'	67° 10.72'	CR
PRBM	Bahia Montalva, Puerto Rico	PR	17° 58.23'	66° 59.43'	CR
PRBJ	Bahia de Jobos, Puerto Rico	PR	17° 56.33'	66° 10.95'	CR
EVFU	Everglades, Faka Union Bay	FL	25° 54.08'	81° 30.78'	CV
RBHC	Rookery Bay, Henderson Creek	FL	26° 01.50'	81° 44.20'	CV
NBNB	Naples Bay, Naples Bay	FL	26° 06.78'	81° 47.15'	CV
CBFM	Charlotte Harbor, Fort Meyers	FL	26° 33.50'	81° 55.37'	CV
CBBI	Charlotte Harbor, Bird Island	FL	26° 30.73'	82° 02.18'	CV
TBCB	Tampa Bay, Cockroach Bay	FL	27° 40.55'	82° 30.56'	CV
TBHB	Tampa Bay, Hillsborough Bay	FL	27° 51.28'	82° 23.75'	CV
TBOT	Tampa Bay, Old Tampa Bay	FL	28° 01.48'	82° 37.95'	CV
TBKA	Tampa Bay, Peter O. Knight Airport	FL	27° 54.46'	82° 27.29'	CV
TBPB	Tampa Bay, Papys Bayou	FL	27° 50.53'	82° 36.62'	CV
TBMK	Tampa Bay, Mullet Key Bayou	FL	27° 37.28'	82° 43.62'	CV
TBNP	Tampa Bay, Navarez Park	FL	27° 47.28'	82° 45.28'	CV
CKBP	Cedar Key, Black Point	FL	29° 12.32'	83° 04.25'	CV
SRWP	Suwannee River, West Pass	FL	29° 19.75'	83° 10.45'	CV
AESP	Apalachee Bay, Spring Creek	FL	30° 03.75'	84° 19.37'	CV
APCP	Apalachicola Bay, Cat Point Bar	FL	29° 43.45'	84° 53.05'	CV
APDB	Apalachicola Bay, Dry Bar	FL	29° 40.45'	85° 04.40'	CV
SAWB	St. Andrew Bay, Watson Bayou	FL	30° 08.53'	85° 37.92'	CV
PCMP	Panama City, Municipal Pier	FL	30° 09.00'	85° 39.80'	CV
PCLO	Panama City, Little Oyster Bar	FL	30° 15.19'	85° 40.95'	CV
			30° 14.27'	85° 42.69'	SED
CBSR	Choctawhatchee Bay, Off Santa Rosa	FL	30° 24.78'	86° 12.25'	CV
CBBL	Choctawhatchee Bay, Bens Lake	FL	30° 27.15'	86° 32.45'	CV
CBPP	Choctawhatchee Bay, Postil Point	FL	30° 28.85'	86° 28.73'	CV
CBBB	Choctawhatchee Bay, Boggy Bayou	FL	30° 30.18'	86° 29.65'	CV
CBJB	Choctawhatchee Bay, Joe's Bayou	FL	30° 24.62'	86° 29.45'	CV
PBSP	Pensacola Bay, Sabine Point	FL	30° 20.80'	87° 09.10'	CV
			30° 21.03'	87° 09.35'	SED
PBIB	Pensacola Bay, Indian Bayou	FL	30° 31.00'	87° 06.70'	CV
PBPH	Pensacola Bay, Public Harbor	FL	30° 24.63'	87° 11.42'	CV
MBDR	Mobile Bay, Dog River	AL	30° 35.50'	88° 02.72'	CV

Table A.46 (cont). Mussel Watch Project sites (Lauenstein *et al.*, 1993).

Site Code	Site name	State	Latitude (N)	Longitude (W)	Species* code
MBHI	Mobile Bay, Hollingers Is. Chan.	AL	30° 33.80'	88° 04.50'	CV
MBCP	Mobile Bay, Cedar Point Reef	AL	30° 18.70'	88° 08.00'	CV
MSPB	Mississippi Sound, Pascagoula Bay	MS	30° 20.03'	88° 36.10'	CV
MSBB	Mississippi Sound, Biloxi Bay	MS	30° 23.55'	88° 51.45'	CV
MSPC	Mississippi Sound, Pass Christian	MS	30° 18.12'	89° 19.62'	CV
MRPL	Mississippi River, Pass A Loutre	LA	29° 04.87'	89° 05.53'	CV
M RTP	Mississippi River, Tiger Pass	LA	29° 08.69'	89° 25.67'	CV
BSSI	Breton Sound, Sable Island	LA	29° 24.11'	89° 29.10'	CV
BSBG	Breton Sound, Bay Gardene	LA	29° 35.90'	89° 37.25'	CV
LBMP	Lake Borgne, Malheureux Point	LA	29° 52.02'	89° 40.70'	CV
LBNO	Lake Borgne, New Orleans	LA	29° 56.60'	89° 50.10'	CV
LPGO	Lake Pontchartrain, Gulf Outlet	LA	29° 52.02'	89° 40.70'	CV
BBTB	Barataria Bay, Turtle Bay	LA	29° 30.67'	90° 05.00'	CV
BBSD	Barataria Bay, Bayou Saint Denis	LA	29° 24.18'	89° 59.75'	CV
BBMB	Barataria Bay, Middle Bank	LA	29° 16.55'	89° 56.53'	CV
TBLF	Terrebonne Bay, Lake Felicity	LA	29° 15.80'	90° 24.40'	CV
TBLB	Terrebonne Bay, Lake Barre	LA	29° 15.60'	90° 35.70'	CV
CLCL	Caillou Lake, Caillou Lake	LA	29° 15.25'	90° 55.80'	CV
ABOB	Atchafalaya Bay, Oyster Bayou	LA	29° 14.40'	91° 08.10'	CV
ECSP	East Cote Blanche, South Point	LA	29° 28.50'	91° 48.00'	CV
VBSP	Vermilion Bay, Southwest Pass	LA	29° 34.60'	92° 02.75'	CV
JHJH	Joseph Harbor Bayou	LA	29° 37.75'	92° 45.75'	CV
CLSJ	Calcasieu Lake, St. Johns Island	LA	29° 49.83'	93° 23.00'	CV
CLLC	Calcasieu Lake, Lake Charles	LA	30° 03.42'	93° 18.42'	CV
SLBB	Sabine Lake, Blue Buck Point	LA	29° 47.50'	93° 54.42'	CV
GBSC	Galveston Bay, Ship Channel	TX	29° 42.27'	94° 59.58'	CV
GBYC	Galveston Bay, Yacht Club	TX	29° 37.30'	94° 59.50'	CV
GBTD	Galveston Bay, Todd's Dump	TX	29° 30.06'	94° 53.82'	CV
GBHR	Galveston Bay, Hanna Reef	TX	29° 28.85'	94° 44.00'	CV
GBOB	Galveston Bay, Offatts Bayou	TX	29° 17.08'	94° 50.15'	CV
GBCR	Galveston Bay, Confederate Reef	TX	29° 15.75'	94° 54.88'	CV
			29° 16.10'	94° 54.60'	SED
BRFS	Brazos River, Freeport Surfside	TX	28° 55.25'	95° 20.33'	CV
BRCL	Brazos River, Cedar Lakes	TX	28° 51.50'	95° 27.83'	CV
MBEM	Matagorda Bay, East Matagorda	TX	28° 42.67'	95° 53.00'	CV
MBDI	Matagorda Bay, Dog Island	TX	28° 38.28'	96° 00.15'	CV
MBTP	Matagorda Bay, Tres Palacios Bay	TX	28° 39.50'	96° 13.45'	CV
MBCB	Matagorda Bay, Carancahua Bay	TX	28° 39.40'	96° 23.18'	CV
MBLR	Matagorda Bay, Lavaca River Mouth	TX	28° 39.80'	96° 34.83'	CV
MBGP	Matagorda Bay, Gallinipper Point	TX	28° 35.25'	96° 34.17'	CV
ESBD	Espiritu Santo, Bill Days Reef	TX	28° 24.85'	96° 26.27'	CV
ESSP	Espiritu Santo, South Pass Reef	TX	28° 17.90'	96° 37.33'	CV

Table A.46 (cont). Mussel Watch Project sites (Lauenstein *et al.*, 1993).

Site Code	Site name	State	Latitude (N)	Longitude (W)	Species* code
SAMP	San Antonio Bay, Mosquito Point	TX	28° 20.65'	96° 42.78'	CV
SAPP	San Antonio Bay, Panther Point Reef	TX	28° 14.00'	96° 42.55'	CV
MBAR	Mesquite Bay, Ayres Reef	TX	28° 10.15'	96° 49.95'	CV
ABLR	Aransas Bay, Long Reef	TX	28° 02.96'	96° 56.77'	CV
CBCR	Copano Bay, Copano Reef	TX	28° 08.47'	97° 07.67'	CV
ABHI	Aransas Bay, Harbor Island	TX	27° 50.33'	97° 04.52'	CV
CCIC	Corpus Christi, Ingleside Cove	TX	27° 50.28'	97° 14.28'	CV
CCNB	Corpus Christi, Nueces Bay	TX	27° 51.17'	97° 21.55'	CV
CCBH	Corpus Christi, Boat Harbor	TX	27° 50.17'	97° 22.72'	CV
LMAC	Lower Laguna Madre, Arroyo Colorado	TX	26° 16.80'	97° 17.30'	CV
LMPI	Lower Laguna Madre, Port Isabel	TX	26° 04.62'	97° 12.05'	CV
LMSB	Lower Laguna Madre, South Bay	TX	26° 02.77'	97° 10.48'	CV
IBNJ	Imperial Beach, North Jetty	CA	32° 35.25'	117° 07.95'	MC
SDCB	San Diego Bay, Coronado Bridge	CA	32° 41.21'	117° 09.53'	ME
SDHI	San Diego Bay, Harbor Island	CA	32° 43.49'	117° 11.68'	ME
			32° 43.14'	117° 11.56'	SED
PLLH	Point Loma, Lighthouse	CA	32° 40.90'	117° 14.92'	MC
			32° 37.00'	117° 15.70'	SED
MBVB	Mission Bay, Ventura Bridge	CA	32° 46.07'	117° 14.47'	ME
LJLJ	La Jolla, Point La Jolla	CA	32° 51.05'	117° 16.15'	MC
			32° 48.75'	117° 19.72'	SED
OSBJ	Oceanside, Municipal Beach Jetty	CA	33° 12.11'	117° 23.56'	ME
			33° 12.80'	117° 28.00'	SED
SCBR	South Catalina Island, Bird Rock	CA	33° 27.10'	118° 29.20'	MC
			33° 26.55'	118° 29.48'	SED
NBWJ	Newport Beach, Wedge Jetty	CA	33° 35.48'	117° 52.77'	MC
			33° 35.12'	117° 53.67'	SED
ABWJ	Anaheim Bay, West Jetty	CA	33° 43.93'	118° 06.02'	MC
			33° 44.27'	118° 07.81'	SED
LBBW	Long Beach, Breakwater	CA	33° 43.42'	118° 10.45'	ME
SPFP	San Pedro Harbor, Fishing Pier	CA	33° 42.42'	118° 16.43'	ME
			33° 42.62'	118° 16.60'	SED
PVRP	Palos Verdes, Royal Palms State Pk.	CA	33° 43.10'	118° 19.35'	MC
			33° 42.65'	118° 21.00'	SED
RBMJ	Redondo Beach, Municipal Jetty	CA	33° 49.91'	118° 23.50'	MC
			33° 49.41'	118° 24.86'	SED
MDSJ	Marina Del Rey, South Jetty	CA	33° 57.68'	118° 27.42'	ME
			33° 59.49'	118° 31.97'	SED
TBSM	Las Tunas Beach, Santa Monica Bay	CA	34° 02.33'	118° 35.85'	MC
			34° 01.60'	118° 33.73'	SED
PDPD	Point Dume, Point Dume	CA	34° 00.08'	118° 48.48'	MC
			33° 59.90'	118° 46.94'	SED
SCFP	Santa Cruz Island, Fraser Point	CA	34° 03.59'	119° 55.25'	MC
SANM	San Miguel Island, Tyler Bight	CA	34° 01.68'	120° 25.16'	MC
SBSB	Pt.Santa Barbara, Pt. Santa Barbara	CA	34° 23.75'	119° 43.72'	MC
			34° 23.15'	119° 43.22'	SED

Table A.46 (cont). Mussel Watch Project sites (Lauenstein *et al.*, 1993).

Site Code	Site name	State	Latitude (N)	Longitude (W)code	Species*
PCPC	Point Conception, Point Conception	CA	34° 26.70'	120° 27.20'	MC
			34° 26.56'	120° 26.00'	SED
SLSL	San Luis Obispo Bay, Point San Luis	CA	35° 09.64'	120° 45.26'	MC
			35° 09.72'	120° 44.12'	SED
SSSS	San Simeon Point, San Simeon Point	CA	35° 38.20'	121° 11.70'	MC
PGLP	Pacific Grove, Lovers Point	CA	36° 37.55'	121° 54.91'	MC
			36° 37.65'	121° 54.15'	SED
MBML	Monterey Bay, Moss Landing	CA	36° 48.09'	121° 47.35'	MC
MBSC	Monterey Bay, Point Santa Cruz	CA	36° 57.20'	122° 01.45'	MC
FIEL	Farallon Island, East Landing	CA	37° 41.77'	122° 59.99'	MC
SFMS	San Francisco Bay, San Mateo Bridge	CA	37° 34.91'	122° 15.16'	ME
			37° 35.30'	122° 13.53'	SED
SFDB	San Francisco Bay, Dumbarton Bridge	CA	37° 30.33'	122° 07.17'	ME
			37° 31.60'	122° 09.63'	SED
SFEM	San Francisco Bay, Emeryville	CA	37° 49.25'	122° 19.70'	ME
			37° 49.69'	122° 20.32'	SED
SPSM	San Pablo Bay, Semple Point	CA	38° 04.20'	122° 14.33'	SED
SPSP	San Pablo Bay, Point San Pedro	CA	38° 01.35'	122° 25.53'	SED
TBSR	Tomales Bay, Spenger's Residence	CA	38° 08.95'	122° 54.17'	ME
			38° 09.03'	122° 54.00'	SED
BBBE	Bodega Bay, Bodega Bay Entrance	CA	38° 18.30'	123° 03.87'	MC
			38° 18.50'	123° 02.84'	SED
PALH	Point Arena, Lighthouse	CA	38° 57.18'	123° 44.30'	MC
PDSC	Point Delgada, Shelter Cove	CA	40° 02.31'	124° 04.76'	MC
			40° 02.38'	124° 04.68'	SED
HMBJ	Eureka, Humboldt Bay Jetty	CA	40° 46.13'	124° 14.25'	MC
			40° 45.06'	124° 12.83'	SED
EUSB	Eureka, Samoa Bridge	CA	40° 49.32'	124° 10.09'	MC
KRFR	Klamath River, Flint Rock Head	CA	41° 31.63'	124° 04.78'	MC
SGSG	Crescent, Point St. George	CA	41° 44.88'	124° 12.52'	MC
			41° 44.25'	124° 11.33'	SED
CBCH	Coos Bay, Coos Head	OR	43° 21.03'	124° 19.85'	MC
			43° 22.17'	124° 18.80'	SED
CBRP	Coos Bay, Russell Point	OR	43° 26.00'	124° 13.15'	ME
			43° 25.75'	124° 13.03'	SED
YHYH	Yaquina Bay, Yaquina Head	OR	44° 40.58'	124° 04.68'	MC
YHSS	Yaquina Bay, Sally's Slough	OR	44° 36.83'	124° 00.95'	SED
YBOP	Yaquina Bay, Oneatta Point	OR	44° 34.98'	124° 00.05'	ME
			44° 34.78'	124° 00.78'	SED
TBHP	Tillamook Bay, Hobsonville Point	OR	45° 32.87'	123° 54.38'	ME
			45° 30.96'	123° 55.59'	SED
CRSJ	Columbia River, South Jetty	OR	46° 14.00'	124° 02.78'	ME
			46° 13.70'	124° 01.12'	MC
CRYB	Columbia River, Youngs Bay	OR	46° 11.00'	123° 52.75'	SED
CRNJ	Columbia River, North Jetty	WA	46° 16.67'	124° 03.73'	ME
			46° 16.15'	123° 59.92'	SED

Table A.46 (cont). Mussel Watch Project sites (Lauenstein *et al.*, 1993).

Site Code	Site name	State	Latitude (N)	Longitude (W)code	Species*
WBNA	Willapa Bay, Nahcotta	WA	46° 29.80'	124° 01.72'	ME
			46° 30.48'	124° 00.36'	SED
GHWJ	Gray's Harbor, Westport Jetty	WA	46° 54.75'	124° 07.05'	MC
			46° 52.55'	124° 04.87'	SED
JFCF	Strait of Juan de Fuca, Cape Flattery	WA	48° 23.30'	124° 43.28'	MC
JFNB	Strait of Juan de Fuca, Neah Bay	WA	48° 22.48'	124° 37.00'	SED
PSPA	Puget Sound, Port Angeles	WA	48° 08.38'	123° 25.01'	ME
			48° 08.28'	123° 25.10'	SED
PSPT	Puget Sound, Port Townsend	WA	48° 06.32'	122° 46.63'	ME
			48° 06.18'	122° 45.90'	SED
PSHC	Puget Sound, Hood Canal	WA	47° 49.90'	122° 41.20'	ME
			47° 50.32'	122° 38.90'	SED
SSBI	South Puget Sound, Budd Inlet	WA	47° 05.94'	122° 53.60'	ME
			47° 06.03'	122° 54.73'	SED
CBTP	Commencement Bay, Tahlequah Point	WA	47° 20.15'	122° 30.10'	ME
CBBP	Commencement Bay, Browns Point	WA	47° 17.58'	122° 25.93'	SED
PSSS	Puget Sound, South Seattle	WA	47° 31.73'	122° 23.92'	ME
			47° 31.55'	122° 24.27'	SED
EBDH	Elliott Bay, Duwamish Head	WA	47° 35.73'	122° 23.20'	ME
			47° 34.55'	122° 25.08'	SED
EBFR	Elliott Bay, Four-Mile Rock	WA	47° 38.35'	122° 24.74'	ME
			47° 37.67'	122° 24.33'	SED
SIWP	Sinclair Inlet, Waterman Point	WA	47° 35.12'	122° 34.15'	ME
			47° 33.05'	122° 37.62'	SED
WIPP	Whidbey Island, Possession Point	WA	47° 54.15'	122° 22.80'	ME
			47° 54.61'	122° 20.64'	SED
PSEH	Puget Sound, Everett Harbor	WA	47° 58.42'	122° 13.72'	ME
			47° 58.43'	122° 14.22'	SED
BBSM	Bellingham Bay, Squalicum Marina Jet.	WA	48° 45.25'	122° 29.97'	ME
			48° 44.77'	122° 30.72'	SED
PRPR	Point Roberts, Point Roberts	WA	48° 59.30'	123° 05.30'	ME
			48° 58.90'	123° 01.30'	ME
			48° 56.47'	123° 00.36'	SED
PVMC	Port Valdez, Mineral Creek Flats	AK	61° 08.17'	146° 27.75'	ME
			61° 06.75'	146° 28.17'	SED
UISB	Unakwit Inlet, Siwash Bay	AK	60° 57.62'	147° 38.67'	ME
			60° 57.35'	147° 39.45'	SED
KAUI	Kauai, Nawiliwili Harbor	HI	21° 57.40'	159° 21.35'	OS
BPBP	Barber's Point, Barber's Point Harbor	HI	21° 19.50'	158° 07.45'	OS
HHKL	Honolulu Harbor, Keehi Lagoon	HI	21° 19.15'	157° 53.30'	OS
			21° 18.15'	157° 53.30'	SED

Table A.46 (cont). Mussel Watch Project sites.

Site Code	Site name	State	Latitude (N)	Longitude (W)code	Species*
GBBS	Green Bay, Bayshore Park	WI	44° 38.20'	87° 47.80'	DP
LMMB	Lake Michigan, Milwaukee Bay	WI	43° 02.00'	87° 53.72'	DP
LMNC	Lake Michigan, North Chicago	IL	42° 18.33'	87° 49.67'	DP
LMNC	Lake Michigan, Calumet Breakwater	IN	41° 43.63'	87° 29.70'	DP
LMNC	Lake Michigan, Holland Breakwater	MI	42° 46.43'	86° 12.88'	DP
LMNC	Lake Michigan, Muskegon Breakwater	MI	43° 13.62'	86° 20.83'	DP
SBSP	Saginaw Bay, Sand Point	MI	43° 54.6'	83° 29.9'	DP
SBSR	Saginaw Bay, Saginaw River	MI	43° 40.59'	83° 50.22'	DP
LHBR	Lake Huron, Black River Canal	MI	43° 02.53'	82° 26.20'	DP
LSAB	Lake St. Clair, Anchor Bay	MI	42° 38.47'	82° 43.12'	DP
LESP	Lake Erie, Stony Point	MI	41° 57.46'	83° 13.58'	DP
LERB	Lake Erie, Reno Beach	OH	41° 40.50'	83° 13.79'	DP
SBPP	South Bass Is., Peach Orchard Point	OH	41° 39.64'	82° 49.45'	DP

\* Species codes

- DP - *Dreissena polymorpha* (zebra mussel)
- ME - *Mytilus edulis* (blue mussel)
- MC - *Mytilus californianus* (Californian mussel)
- CR - *Crassostrea rhizophora* (mangrove oyster)
- CS - *Chama sinuosa* (smooth edge-jewel box)
- CV - *Crassostrea virginica* (American oyster)
- OS - *Ostrea sandvicensis* (Hawaiian oyster)
- SED - Sediment site location

Sites in Green Bay and Lake Michigan were first collected in 1993, while the other Great Lake sites were first collected in 1992.

